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PATENT COOPERATION TREATY

PCT

NOTIFICATION OF ELECTION

(PCT Rule 61.2)

From the INTERNATIONAL BUREAU

To:

Commissioner
 US Department of Commerce
 United States Patent and Trademark
 Office, PCT
 2011 South Clark Place Room
 CP2/5C24
 Arlington, VA 22202
 ETATS-UNIS D'AMERIQUE
 in its capacity as elected Office

Date of mailing (day/month/year) 11 April 2001 (11.04.01)	
International application No. PCT/BR00/00065	Applicant's or agent's file reference PE-3849
International filing date (day/month/year) 23 June 2000 (23.06.00)	Priority date (day/month/year) 23 June 1999 (23.06.99)
Applicant GARCIA PINATTI, Daltro et al	

1. The designated Office is hereby notified of its election made:

☒ in the demand filed with the International Preliminary Examining Authority on:
 22 January 2001 (22.01.01)

☐ in a notice effecting later election filed with the International Bureau on:

2. The election ☒ was
☐ was not

made before the expiration of 19 months from the priority date or, where Rule 32 applies, within the time limit under Rule 32.2(b).

The International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland Facsimile No.: (41-22) 740.14.35	Authorized officer Olivia TEFY Telephone No.: (41-22) 338.83.38
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International Bureau(43) International Publication Date
28 December 2000 (28.12.2000)

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WO 00/78446 A3(51) International Patent Classification⁷: B01J 19/02.
19/28, C13K 1/02

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PI 9902607-4 23 June 1999 (23.06.1999) BR

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(72) Inventors; and

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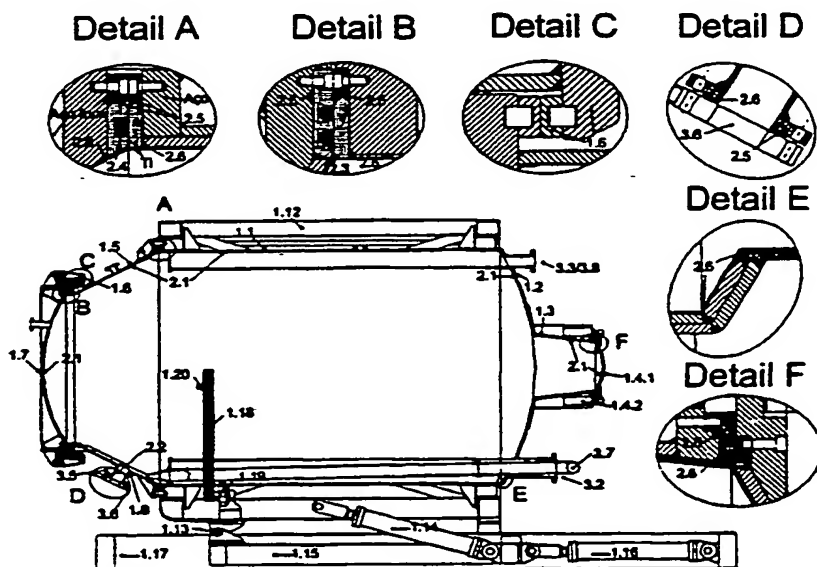
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(81) Designated States (national): AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CR, CU, CZ, DE, DK, DM, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW.

(84) Designated States (regional): ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European

[Continued on next page]

(54) Title: AN APPARATUS AND PROCESS FOR PRE-HYDROLYSIS OF BIOMASS



(57) Abstract: This invention refers to an apparatus for the processing of pre-hydrolysis of biomass, particularly a reactor of the failsafe type, either mobile or not, which may be manufactured of microbonded carbon steel and coated with special materials (refractory metals, superalloys, stainless materials, plastics, etc.). The insertion of vacuum between the shell of the reactor and its coating prevents its implosion and permits the detection of leakage with helium gas. The resistance to corrosion of the refractory metals enables carrying out of pre-hydrolysis and mineral digestion. In the process of this invention, biomass is compacted at 300 kg/m³ by a helical feeder, degased, flooded, heated, stirred, pre-hydrolyzed, the sugar being recovered at a first washing within the reactor with values higher than 90 %.

The low liquid/solid ratio = 2 enables one to achieve sugar content of 10 bricks in the pre-hydrolysate and a consumption of 6 % of the energy contained in the processed biomass. The pre-hydrolysate is constituted by solutions of xylose and others and is intended for the production of furfural, alcohol and xylitol. The control of the process enables one to produce cellulignin with a high specific surface, 2 m²/g (measured by BET), compared with the value of 0.4 m²/g of non-hydrolyzed biomass, maintain crystallinity of the cellulose fibers, aiming at grinding it into fine particles (f<250 μm), globulizing the lignin, exposing the cellulose to attacks by microorganisms from cud of ruminants, fungi, bacteria or enzymes in microbiological or enzymatic fermentation. The catalytic combustion due to the large specific surface results in a new fuel for boilers, combustors in general and MHD when produced with filtered water, and for gas turbines when produced with deionized water.



patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE). OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

(88) Date of publication of the international search report:
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For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 B01J19/02 B01J19/28 C13K1/02

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 B01J C13K F16L F17C

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, WPI Data, PAJ

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 3 056 664 A (DRAVNIKS ANDREWS, FOREST. PARK, TROSCINSKI EDWIN S, BIRKNESS HARALD A) 2 October 1962 (1962-10-02) the whole document	1,6,7,9
P,X	WO 00 31459 A (STEYR DAIMLER PUCH AG ;BRUNNHOFER KLAUS (AT)) 2 June 2000 (2000-06-02) the whole document	1,5,9
X	US 4 997 124 A (KITABATAKE AKIHIRO ET AL) 5 March 1991 (1991-03-05) column 5, line 9 - line 21 column 6, line 22 - line 42 column 8, line 56 -column 9, line 20 figures 1,2,9,10 -/-	1,5

☒ Further documents are listed in the continuation of box C.☒ Patent family members are listed in annex.

* Special categories of cited documents:

- *A* document defining the general state of the art which is not considered to be of particular relevance
- *E* earlier document but published on or after the international filing date
- *L* document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- *O* document referring to an oral disclosure, use, exhibition or other means
- *P* document published prior to the international filing date but later than the priority date claimed

- *T* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
- *X* document of particular relevance: the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
- *Y* document of particular relevance: the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
- *S* document member of the same patent family

Date of the actual completion of the international search

5 July 2001

Date of mailing of the international search report

12 07 2001

Name and mailing address of the ISA

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Authorized officer

Lepretre, F

INTERNATIONAL SEARCH REPORT

International Application No.

PCT/BR 00/00065

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	WO 99 26720 A (ALLIED SIGNAL INC) 3 June 1999 (1999-06-03) page 5, line 1 -page 6, line 11 page 7, line 9 - line 21 page 8, line 12 -page 9, line 14 claims 1-16; figure 1	1,6,7, 9-14
A	US 5 159 694 A (OVERATH HORST, SOEDER CARL-JOHANNES, SALHANI NAZIR) 27 October 1992 (1992-10-27) column 5, line 65 -column 6, line 12 figure 4	1-4,6, 15-25
A	GB 1 569 138 A (VALKANAS ET AL.) 11 June 1980 (1980-06-11) claims; example 1	15-25
A	US 4 199 371 A (REGNAULT ET AL.) 22 April 1980 (1980-04-22) claims; examples	15-25

INTERNATIONAL SEARCH REPORT

...ternational application No.
BR 00/00065

Box I Observations where certain claims were found unsearchable (Continuation of item 1 of first sheet)

This International Search Report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. ☐ Claims Nos.:
because they relate to subject matter not required to be searched by this Authority, namely:
2. ☐ Claims Nos.:
because they relate to parts of the International Application that do not comply with the prescribed requirements to such an extent that no meaningful International Search can be carried out, specifically:
3. ☐ Claims Nos.:
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).

Box II Observations where unity of invention is lacking (Continuation of item 2 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

see additional sheet

1. ☒ As all required additional search fees were timely paid by the applicant, this International Search Report covers all searchable claims.
2. ☐ As all searchable claims could be searched without effort justifying an additional fee, this Authority did not invite payment of any additional fee.
3. ☐ As only some of the required additional search fees were timely paid by the applicant, this International Search Report covers only those claims for which fees were paid, specifically claims Nos.:
4. ☐ No required additional search fees were timely paid by the applicant. Consequently, this International Search Report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:

Remark on Protest

- ☐ The additional search fees were accompanied by the applicant's protest.
- ☒ No protest accompanied the payment of additional search fees.

FURTHER INFORMATION CONTINUED FROM PCT/SA/ 210

This International Searching Authority found multiple (groups of) inventions in this international application, as follows:

1. Claims: 1-14

Independent claim 1 discloses an apparatus comprising an external and an internal coating and a space between the two coatings adapted to be under vacuum. The dependent claims disclose further details of the apparatus concerning an oscillation mechanism, the pressure within said space, the materials and the way of mounting of the coatings and a leakage detection system.

2. Claims: 15-25

Independent claim 15 discloses a process of biomass pre-hydrolysis characterized in that said process is carried out with a rotary oscillation apparatus. The dependent claims disclose further details of said process.

INTERNATIONAL SEARCH REPORT

Information on patent family members

Invention Publication No
PCT/BR 96/00065

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
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INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/BR 00/00065

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
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International Bureau



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PCT

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PI 9902607-4 23 June 1999 (23.06.1999) BR

(71) Applicant (for all designated States except US): RM MATERIAIS REFRATÁRIOS LTDA. (BR/BR); Estrada do Pinhal, 750, CEP-12600-000 Lorena, SP (BR).

(72) Inventors; and

(75) Inventors/Applicants (for US only): GARCIA PINATTI,

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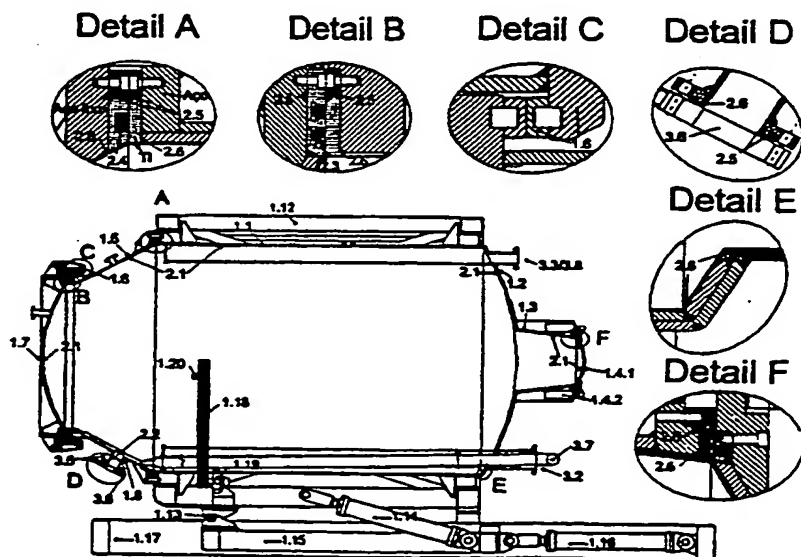
(74) Agent: DANNEMANN, SIEMSEN, BIGLER & IPANEMA MOREIRA; Caixa Postal 2142, Rua Marquês de Olinda, 70, Botafogo, CEP-22251-040 Rio de Janeiro, RJ (BR).

(81) Designated States (national): AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CR, CU, CZ, DE, DK, DM, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW.

(84) Designated States (regional): ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European

[Continued on next page]

(54) Title: AN APPARATUS AND PROCESS FOR PRE-HYDROLYSIS OF BIOMASS



(57) Abstract: This invention refers to an apparatus for the processing of pre-hydrolysis of biomass, particularly a reactor of the failsafe type, either mobile or not, which may be manufactured of microbonded carbon steel and coated with special materials (refractory metals, superalloys, stainless materials, plastics, etc.). The insertion of vacuum between the shell of the reactor and its coating prevents its implosion and permits the detection of leakage with helium gas. The resistance to corrosion of the refractory metals enables carrying out of pre-hydrolysis and mineral digestion. In the process of this invention, biomass is compacted at 300 kg/m³ by a helical feeder, degased, flooded, heated, stirred, pre-hydrolyzed, the sugar being recovered at a first washing within the reactor with values higher than 90 %.

The low liquid/solid ratio = 2 enables one to achieve sugar content of 10 bricks in the pre-hydrolysate and a consumption of 6 % of the energy contained in the processed biomass. The pre-hydrolysate is constituted by solutions of xylose and others and is intended for the production of furfural, alcohol and xylitol. The control of the process enables one to produce cellulignin with a high specific surface, 2 m²/g (measured by BET), compared with the value of 0.4 m²/g of non-hydrolyzed biomass, maintain crystallinity of the cellulose fibers, aiming at grinding it into fine particles (f<250 μm), globulizing the lignin, exposing the cellulose to attacks by microorganisms from cud of ruminants, fungi, bacteria or enzymes in microbiological or enzymatic fermentation. The catalytic combustion due to the large specific surface results in a new fuel for boilers, combustors in general and MHD when produced with filtered water, and for gas turbines when produced with deionized water.



patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

Published:

— *Without international search report and to be republished upon receipt of that report.*

Title: "AN APPARATUS AND PROCESS FOR PRE-HYDROLYSIS OF BIOMASS"**Field of the invention**

This invention relates to an apparatus, specifically a reactor, and to a process for pre-hydrolysis of any kind of biomass (wood, bagasse, straw, grass, vegetable residues, organic garbage, etc.) to obtain commercial products, such as cellulignin and sugar solutions, pre-hydrolysate layers, containing xylose, glucose, mamose, galactose, arabinose, acethyl-ics, uranic acids, etc. This invention further enables one to manufacture reactors for the di-gestion of minerals, such as ilmenite, anatasie, columbite, pyrochlore, zirkonite, etc.

Description of the prior art

10 Chemical reactors are devices that are defined by a variety of processes for transforming materials, such as chemical, metallurgical, thermal, biotechnological, pharma-ceutical materials, etc. Reactors are classified by three criteria:

15 a) classification according to vessels and flows: 1st) batch reactor; 2nd) semi-continuous reactor; 3rd) tank reactor with continuous mixer – RTMC; 4th) RTMC in series; 5th) tubular reactor; and 6th) recycling reactor.

20 b) classification according to the existing phases: 1st) reactors for gaseous phases; 2nd) reactors for liquid phases; 3rd) reactors for liquid and gaseous phases; 4th) reactors with solid catalysts; 5th) non-catalytic reactors involving solids (gas-solid, liquid-solid, and reactions of gaseous phases; 6th) reactors for electrochemical processes; 7th) reactors for biological processes; 8th) reactors for photochemical and radiochemical proc-esses. The non-catalytic gas-solid and liquid-solid reactors with which, by way of example, the reactor of this invention should be compared, are listed below.

b.1) non-catalytic gas-solid reactor: retort (tower-type reactor), movable bed reactor, multiple bed reactor, rotary oven reactor, fluid bed reactor, recirculating flow reactor, dust calciner (dust spray).

b.2) non-catalytic liquid-solid reactors: tank with a mixer (batch, semi-continuous, cascade - percentage of solids limited by the power of the mixer), tank with recirculation of liquids, rotary drum, fluid bed, kneading machine, immersion-press type reactor, helical conveyor, cradle-type reactor, rotary oven reactor.

c) Classification according to the movement condition of the solids presented in Table 1. The reactor of this invention tends towards the characteristics of a fluid bed, albeit movable, with the following values: reaction device (tank), movement of the solids (gravitational), gas/liquid-solid flow (counter current), particle diameter (mm to cm), time of retention of the solids (minutes), time of retention of the gas (seconds), rate of heat and mass transfer (high), temperature control (good), yield in terms of volume and time (high).

Other important characteristics of reactors are the materials used to manufacture them. Table 2 gives the basic materials with their respective advantages and disadvantages for high corrosion conditions. The structural materials and those resistant to corrosion, even when used in making reactors, each presents a single disadvantage, which this invention eliminates by making the reactor with a shell and structure made of carbon steel and a coating of special materials (refractory, superalloys, stainless steel and plastics, etc.).

Specifically, the main biomass reactors are of the following types: screw (low volume occupation percentage), tower (high L/S - liquid-solid ratio, tank/percolation (high L/S ratio and plug flow (high L/S ratio). All the types of reactor have complex biomass feed and discharge systems. This invention compacts biomass in the reactor (high rate of volume occupation of the reactor), processes the biomass with low L/S ratio = 2, provides feed with a simple helical feeder and immediate discharge due to the fact that the reactor has a large cover and can be divided into small-size and medium-size reactors.

One disadvantage of the reactors of the prior art is that they are subject to implosion due to the fact that their construction does not permit a perfect juxtaposition between the shell and the coating, and there is a vacuum inside the reactor, caused, in most cases, by steam condensation. This invention prevents implosion through the vacuum maintained between the coating and the shell, so as to allow them to be properly juxtaposed.

Another disadvantage of the conventional reactors is the impossibility of continuously and precisely detecting microleakage in the coating. This invention permits the precise detection of microleakage before the occurrence of leakage of corrosive liquids into the steel shell of the reactor. This allows the reactor to be further used, even with microfailures, by adequately programming it for use.

The reactors used in high-corrosion applications do not utilize carbon steel in the shell and tend to be made of expensive monolithic materials. By enabling the identification of microleakages, this invention detects the conditions for accelerated corrosion of the microbonded carbon steel, and this detection facilitates the use thereof, which brings about a significant reduction in the costs of manufacturing the reactor.

The reactors of the prior art are built from thick materials, in order to achieve greater resistance to corrosion. Therefore, sometimes the thickness of the metal in the construction of the reactors has a direct relationship with the safety, and the utilization of very thick plates makes their transportation to areas where there is raw material for their use impossible. The reactor of this invention provides perfect control for detection of failures and allows lighter materials to be used, which reduces the total weight of the reactor and consequently facilitates its transportation.

US Pat. 5,338,366 refers to a method and apparatus for pre-hydrolysis of biomass that, among other features that are different from this invention, is very heavy and so remains in a stationary position, which requires raw biomass to be transported to it. In addition, the pre-hydrolysis of biomass is made in various pieces of equipment, which increases the consumption of energy for carrying it out and renders the production process expensive. This invention, in addition to eliminating these drawbacks, facilitates a high concentration of sugar in the pre-hydrolysate, thus reducing the costs of the following processes, and enables one to use deionized water due to the low liquid/solid ratio for the production of clean catalytic cellulignin fuel for use in gas turbines.

US Pat. 5,411,594 deals with the hydrolysis of lignocellulose biomass for the production of a single sugar solution and lignin residues. The whole hydrolysis is carried out in two stages (hemicellulose and cellulose) in a continuous reactor and has an L/S ratio of 5:10 with a maximum output of 65%, and, due to the high L/S ratio, there is a complex heat-recovery system. This invention implements the whole process, since it carries out the pre-hydrolysis of hemicellulose in a batch-type reactor with an L/S ratio = 2 and has an output of 100% (maximum utilization of raw material): recovery of cellulignin 80% and pre-hydrolysate

20%. The recovery of heat takes place only when discharging the pre-hydrolysate, pre-heating the acidic solution and water in the boiler.

Table 1 – Classification according to the state of movement of the solids


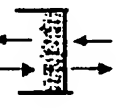

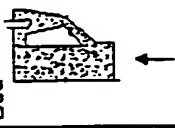
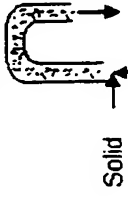
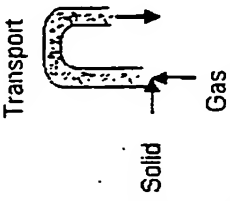
Reactor type	Fixed bed		Fluid bed		Transport
	Upper flow 	Permeable 	Fluid Bed 	Expanded Fluid Bed 	
Typical reaction devices	Multiple cradle Rotary furnace Drying belt	Vertical furnace Movable grid Burning on grid Furnaces	Fluid roaster Fluid bed roaster Multi-stage Fluid bed Furnace	Circulating fluid bed	Flash drying Pre-heater Fusion cyclone Burner
Movement of solids	Mechanical	Gravitational	Gravitational		Gravitational
Gas/solid flow	Parallel stream Countercurrent Crossed current		Mixed flow Countercurrent in stages Crossed current		Parallel currents Mixed flow: recycling Stages in counter-current
Particle diameter	Small to very large (mm/m)	Medium to very large (cm/m)	Small and medium (mm/cm)	Very small to small (mm/mm)	Very small (mm)
Retention time of the solids	Hours - days		Hours	Minutes	Seconds and fractions
Retention time of the gas	Seconds		Seconds		Fractions of seconds

Table 1 - cont.

Reactor type	Fixed bed			Fluid bed			
	Very low	Low – medium	High	Very high	Very high	Very high	
	Very good	Poor – medium	Good	Very good	Medium - good	Medium - good	
	Very low	Medium	Medium High	High	Very high	Very high	
Transfer rate of heat and mass	Very low	Low – medium	High	Very high	Very high	Very high	
Temperature control	Very good	Poor – medium	Good	Very good	Medium - good	Medium - good	
Output in volume and time	Very low	Medium	Medium High	High	Very high	Very high	

Tabl 2 – Basic Mat rials Used in th Manufacture of th Reactors

MATERIALS		ADVANTAGES	DISADVANTAGES
Structural mat rials	Mo (C-1/2Mo) carbon steel, Cr-(1 ¼ Cr- ½ Mo, 2 ½ Cr – 1Mo, 5 Cr-1Mo) steel, stainless (405, 410, 304, 316, 321, 347, 309, 310, steel resistant to heat (HH, HK, HP), monel, brass, bronze, aluminum, etc.	<ul style="list-style-type: none"> ·Low cost ·Availability ·Resistance to abrasion ·Ease of manufacture ·Mechanical resistance (strength) to medium temperature ·Recyclable ·Ductility 	<ul style="list-style-type: none"> ·Low resistance to corrosion
Metals resis- tant to corro- sion	Cr-Mo (9Cr-½ Mo), steel, inconel, incoloy, hastelloy, duplex allows, Fe-Si, stainless, ferritic, Ni (3½ Ni, etc.) steels, lead, Cu-Ni (70/30, 90/10, etc.), re- fractory metals (Ti, Zr, Nb, Ta, W, Mo and their alloys) and proprietary alloys	<ul style="list-style-type: none"> ·Medium and high resistance to corrosion ·Availability ·Resistance to abrasion ·Ease of manufacture ·Mechanical resistance to high temperature ·Recyclable ·Ductility (except for Fe-Si). 	<ul style="list-style-type: none"> ·High cost
Thermoplastic materials	Teflon (260°C), chlorinated polyester (121°C), polyvi- nylidene fluoride (121°C), polyvinylidene chloride (93°C), polypropylene PP (90°C), polyethylene PE (93°C), polyvinyl chloride – PVC (71°C), chlorinated polyvinyl chloride and re- inforced PVC (99°C)	<ul style="list-style-type: none"> ·Low cost ·Availability ·Ease of manufacture ·High resistance to corrosion ·Recyclable ·Ductility 	<ul style="list-style-type: none"> ·Low resistance to abrasion ·Low mechanical resistance (strength)
Elastomers	Natural rubber (66°C), ebonite (82°C), hypalon (93°C), neoprene (93°C), buthylic rubber (93°C), hycar (121°C), viton (204°C).	<ul style="list-style-type: none"> ·Low cost ·Availability ·Ease of manufacture ·High resistance to corrosion ·Ductility 	<ul style="list-style-type: none"> ·Low resistance to abrasion at medium temperature ·Low mechanical resistance (strength) ·Non-recyclable
Reinforced heat cured plastics	Glass fiber/polyester (93°C), asbestos/epoxy (149°C), glass fiber glass/epoxy (149°C), as- bestos/fenolic (149°C), asbestos/furanic (149°C), glass fiber/furanic (93°C), carbon fiber/furanic (93°C).	<ul style="list-style-type: none"> ·Availability ·High resistance to corrosion ·Ease of manufacture ·High mechanical resistance (strength) ·Ductility 	<ul style="list-style-type: none"> ·High cost ·Low resistance to abrasion ·Non-recyclable
Special non- metallic mate- rials	Cement or concrete, rein- forced cement, antiacid refractories, graphite bricks, impermeable graphite, glasses and vitri- fication, wood and refracto- ries	<ul style="list-style-type: none"> ·Low cost ·Availability ·High resistance to abrasion ·Ease of manufacture ·High resistance to corrosion ·Mechanical resistance to high temperature 	<ul style="list-style-type: none"> ·Low mechanical resistance (strength) ·Fragility ·Non-recyclable

Summary of the invention

This invention refers to an apparatus, more specifically a reactor or boiler, built with technology that enables use in highly corrosive environments. The new technology consists of inserting a vacuum between its outer coating, the "shell", and the inner coating, so as to utilize microbonded carbon steel in the shell and structure of the reactor, and special materials, especially refractory metals (Ti, Zr, Nb, Ta, and their alloys) for the inner coating. The vacuum with the coating of refractory material further permits the use of high-resistance (750 MPa) microbonded steel in the manufacture of the ultralight shell of the "egg-shell"-type reactor, enabling oscillation at higher frequencies with higher homogenization of the product and shorter reaction time thereof.

The satisfactory vacuum level is below 1mm of Hg (1,000 μm), so that the refractory metal coating will not suffer implosion, and so that it is possible to use a helium leakage detector coupled between the reactor and the vacuum pump for detecting and measuring leakage through the microcracks of the welds. Leakage rates of 10^{-4} Ncm³/min indicate a satisfactory level and leakage rates 100 times higher (10^{-2} Ncm³/min) indicate the need for repair of the welding cracks or those developed during operation of the reactor. Another characteristic of the invention is the possibility of maintaining and maybe replacing the coating of refractory metal altogether, with high aggregated value for recovery of these scrapped metals due to the carbon steel reactor's juxtaposed manufacture, maintained by the vacuum and without welding to the shell.

The vacuum between the shell of the reactor and the coating maintains the thin coating of special metal juxtaposed to the steel shell, preventing the implosion of the reactor, even when there is vacuum inside it, due to the condensation of steam and other causes. The use of vacuum, among other advantages, also permits continuous or intermittent detection of microleakage in the coating of the reactor by injecting helium gas into the reactor and detecting with a leak detector) in the vacuum pump.

Due to this characteristic, this invention is called "failsafe-type reactor", by means of which one detects and measures microleakage in periods of days or months before the occurrence of leakage of corrosive liquids into the shell of the carbon steel. This characteristic enables one to use the reactor even with microfailures, and to program its repair or replacement after the industrial session or campaign has finished. One should note the significance of this characteristic of the invention for enabling one to use carbon steel in the re-

actor shell for high-corrosion applications, without which leakage and consequently accelerated corrosion of the microbonded carbon steel would not be identified.

The apparatus of this invention, in being lighter than those of the prior art, permits oscillation during its hydrolysis process, which allows the limit-layer of the sugar-saturated liquid to be renewed on the surface of the biomass chips or particulate, thus bringing about a homogeneous product. Pre-hydrolysis coupled with oscillation and purging of steam will provide the characteristic of catalytic combustion of the cellulignin.

Objectives of the invention

One objective of the invention is to provide the use of carbon steel in processes carried out in highly corrosive mediums.

Another objective of this invention is to identify corrosion of carbon steel used in the reactor.

Another objective of this invention is to prevent implosion of the reactor, even when there is a vacuum inside it.

Another objective of this invention is to permit detection of leakage or even microleakage in reactors.

Another objective of this invention is to enable one to program periods of maintenance or repair of the reactor adequately.

Another objective of this invention is to reduce the weight of the reactor and to enable one to transport it to places where there are raw materials.

Another objective of this invention is to reduce the costs of manufacturing the reactor by virtue of the possibility of using cheaper materials.

Another objective of this invention is to enable one to use a more resistant steel as shell/outer coating, so as to permit oscillation at higher frequencies, in order to better homogenize the product and shorten its reaction time.

Another objective of this invention is to meet the requirements of Resolution CONAMA No. 20 of June 18, 1986, published in the D.O.U. (Official Gazette) of July 30, 1986, in washing waters, imparting to the invention totally ecological features.

Brief description of the figures

Figure 1 shows a flowchart of the pre-hydrolysis of biomass with balance of mass.

Figure 2^a shows an embodiment of the reactor in cross-section and its manufacture details.

Figure 2b is a side view showing the placing of an embodiment of the reactor on a transporting cart.

Figure 2c is a top view of an embodiment of the reactor with a feeder.

Figure 2d is a rear view of an embodiment of the reactor on the transporting cart.

Figure 2e is a front view of an embodiment of the reactor positioned on pillars in the biomass store with the cellulignin-unloading cart

Figure 3 shows microphotos of cellulignin (pre-hydrolyzed eucalyptus), crystalline cellulose and globulized lignin.

Figure 4 is a graph showing the average specific power irradiated during the combustion of a cellulignin particle.

Figure 5 is a graph showing the electrical conductivity of the combustion plasma for gas, heavy oil, coal, tar, and cellulignin.

Detailed description of the figures and of the characteristics of the invention

The flowchart of biomass processing is shown in figure 1, where the "failsafe type reactor" of this invention is the main equipment. In the processing of minerals, the steps are similar, only there is adaptation of the peripheral pieces of equipment and adjustment of the mass balance for each type of mineral. The flowchart is composed of the following steps:

1) feeding platform - where the biomass (or mineral) is discharged and washing is carried out or not;

2) helical feeder - responsible for feeding and compacting biomass in the reactor (this compaction is dispensable in the case of minerals);

3) reactor - main object of the invention, where the processes of biomass hydrolysis, mineral digestion or any other chemical or thermochemical processes of any material are developed;

5 4) heat-exchanger - having three functions, namely: a) recovery of the heat while discharging the pre-hydrolysate, b) pre-heating of the acidic solution, and c) pre-heating of the steam-generating water in the boiler;

10 5) tank or washing cart - where the cellulignin (or mineral) is washed to the desired extent, the pre-washing being recovered as pre-hydrolysate and the other washings being neutralized and treated in a stabilization pond prior to discharge into streams or for use in fertilization, etc.;

6) tank of pre-hydrolysate, either stationary or installed on carts - storing of the first product;

7) cellulignin or mineral dryer - drying of the second product;

8) cellulignin or mineral grinder - grinding of the second product;

15 9) silo, either stationary or installed on carts - storing of the second product.

The invention defines a new technology of manufacturing chemical reactors for processes in highly corrosive environments. Figure 2 illustrates in detail a reactor built with the technology of this invention and, in addition, shows all the components of an ordinary reactor for better understanding.

20 The conditions of internal corrosion of the reactor for different materials, temperature and acid content are given in Table 3, whence the following conclusions are drawn:

1) due to its high corrosion rate, stainless steel can only be used at high temperatures for a very short period of time; 2) it is possible to use commercial-grade Ti for the conditions of pre-hydrolysis of biomass ($T = 15^{\circ}\text{C}$, 1.5% H_2SO_4 , 0.62 MPa), since this is a reactive material and passivates with sugar generated by the hydrolysis itself; 3) for the conditions of total hydrolysis of biomass ($T = 190^{\circ}\text{C}$, 1.5% H_2SO_4 , 2.8 MPa) it is necessary to use NbTi alloys (example: Nb44%Ti) with a melting point $T = 1,830^{\circ}\text{C}$ permitting easier welding than pure Nb, which has a melting point of $T = 2,500^{\circ}\text{C}$; 4) for the conditions of mineral digestion ($T = 150^{\circ}\text{C}$, 60% H_2SO_4 , $P = 0.6\text{ MPa}$), it is necessary to use Nb40%Ta alloy, the corrosion rate

25

of which is in the order of 100 mm/year. Above 40% Ta, the alloys become expensive due to the need for using tantalum mineral instead of the columbite in the production of NbTa alloy.

Table 3 - Corrosion Data for the Conditions of Pre-hydrolysis, Hydrolysis of Biomass and Mineral Digestion (mm/year)

	Biomass			Mineral		
		Pre-hydrolysis	Hydrolysis	Mineral digestion		
T°C X% H ₂ SO ₄	100°C 1%	150°C 1.5%	190°C 2%	143°C 60%	197°C 80%	Cost of material. US\$/kg
SS 316	3,550	∞	∞	∞	∞	8
Ti	2,500	19,000	∞	∞	∞	50
Hastelloy	25	—	250	> 5,000	∞	40
Nb	0	4.5	< 25	574	31,761	50
Nb20%Ta	—	—	< 1	367	19,243	50
Nb40%Ta	—	—	—	67	4,093	50
Nb60%Ta	—	—	—	22	916	3,200
Nb80%Ta	—	—	—	3.4	275	410
Ta	0	< 1	< 1	0.1	111	500
Ti 1% H ₂ SO ₄ 7.2% Wood		100				

5

The details of figure 2 present the method of welding the refractory metal coating *in situ* and juxtaposed to the shell of the carbon steel reactor, without the former becoming molten or contaminating the refractory metal. Protection rings or plates of the same metal as the coating are placed on the welding of the parts and edges. The welding is carried out with plasma with inert-gas protection (helium or argon), guaranteeing interstice levels (C, O, N, H) within the norms of the initial material. As to the metallurgical characteristics, there is a growth of grains in the welded zones, decreasing the strength, mainly the fatigue strength. This decrease is acceptable, since the coating works in compression regime, and there is an increase in the coating in regions under traction regime. The construction of the reactor having a bipartite structure with a slight conicity at the cylindrical parts allows much of the welding to be carried out outside the carbon steel shell and only the junctions of large parts and edges to be welded *in situ*. It is also possible to recover significant portions of the coating, in case it is necessary to replace it, by virtue of the ease of disassembling it, thus recovering part of its cost as scrap of high value. The special metals enable the use of other acids, in addition to H₂SO₄ (HCl, HNO₃, etc.), provided that the effective pH is similar to the levels of the solutions of Table 3.

20

Another feature of this invention is the use of elastomer rings (voton, silicone, teflon, and others) having a circular or trapezoidal cross-section on all the covers, connections and vacuum sealing in the coating. The sizing of the sealing rings follow standards of pressure and vacuum sealing. The type of ring is chosen in function of the temperature.

- 5 When processing minerals or biomass, the temperature usually is around 200° C, and the ring chosen is made of viton. On the other hand, for average temperatures, the ring should be of teflon/glass fiber/carbon fiber, the metallic one being most recommended for high temperatures.

- 10 In addition to biomass and minerals, it is possible to process any type of material in the "failsafe type reactor" (polymerization, foodstuffs, medicaments, synthesis of products, etc.), provided that the conditions of corrosion of the coating are respected. In highly aggressive conditions, one uses Ta, either pure or doped with platinum, which are insuperable materials compared with any other commercial material. For less aggressive conditions, the "failsafe" technique permits the use of lower quality noble coatings, mentioned in Table 2
15 (stainless materials, nickel alloys, thermoplastics, elastomers, reinforced thermocured plastic, etc.), provided that the conditions of temperature, pressure, corrosion, abrasion, and strength of the material are respected.

- 20 The external conditions of corrosion of carbon steel are more aggressive at the moment of discharge of the products (cellulignin, minerals, etc.). Steams were condensed at the moment of discharge, their pH was measured, and various steels painted with bituminous epoxy paint were tested.

Table 4 gives the comparison of the costs/m² of the reactors coated with Ti, Nb and monolithic made of stainless steel and hastelloy. In addition to the technical superiority, they have lower costs than the monolithic reactors.

25 **Table 4 – cost of the materials used in the construction of the reactor/m²**

Coating Type	Material	Total Weight	Price/kg	Total Price	Final Price R\$/m ²
Titanium	Steel plate 12 mm	91 kg	R\$ 1,00	R\$ 91,00	R\$ 379,00
	Ti plate 2 mm	9 kg	R\$ 32,00	R\$ 288,00	
Niobium	12 mm steel plate	91 kg	R\$ 1,00	R\$ 91,00	R\$ 1.211,00
	Nb plate 2 mm	16 kg	R\$ 70,00	R\$ 1.120,00	
Stainless steel	12 mm plate	91 kg	R\$ 16,00	R\$ 1.456,00	R\$ 1.456,00
Hastelloy	12 mm plate	91 kg	R\$ 32,00	R\$ 2.912,00	R\$ 2.912,00

The main characteristics of this invention are shown in figure 2, and Table 5 gives the caption of the parts and components of the reactor and of the feeder.

The reactor body is composed of the cylindrical portion 1.1, cap 1.2, inlet nozzle 1.3, inlet cover 1.4, conical sector 1.5, circular or radial sliding ring with wedges or pins 1.6, discharge cover with support of the wedge 1.7, valve connections, meters, accessories, etc. 1.8, hinge 1.9, articulation and hydraulic cylinder for closing the cover 1.10, hydraulic cylinder for closing and opening the wedges 1.11. The bipartition of the reactor is illustrated in detail A, which enables the assembly and disassembly of the special metal coating without welding or unwelding the steel shell of the reactor. For small reactors, the cover is displaced for bipartition, the conical sector being eliminated (for very small reactors, the shell and the coating may be juxtaposed only by fitting, without the need for welding). The cylindrical body is slightly conical to facilitate the assembly and disassembly of the coating. The latter is welded, in great part, prior to the assembly, the parts shown in details A - F remaining to be welded within the reactor shell. This technology is fundamental to make possible the welding close to the steel body of the reactor (melting point of 1760°) of the special metals having a high melting point ($1800^{\circ}\text{C} - 3100^{\circ}\text{C}$), preventing the contamination thereof with Fe. The opening and closing of the cover is done by means of two hydraulic systems, the first one commanding the internal-pressure support wedges of the reactor, maintaining the confinement pressure of the sealing ring (o-ring) of viton of the acidic solutions. This sealing ring supports for months or years the repetitive opening and closing during the operations.

The tilting system is composed of structure 1.12, pivot 1.13, and hydraulic cylinder 1.14. The latter is utilized to tip and shake the reactor during the operation of discharging the cellulignin, since it is a solid material. The horizontal motion system is composed of a cart 1.15 and a hydraulic cylinder 1.16. Alternately, the reactor may be fixed and the feeder may be horizontally moved. The fixed structure 1.17 supports the reactor and the feeder, and thereby the reactor can be detached from the cart to the support pillars (see figure 2.d). The turning oscillation system is composed of chain 1.18 and a hydraulic motor 1.19 commanded by micro-switches 1.20, which revert the rotation at pre-determined angles. The security of the turning oscillation system is given by the length of the chain, which escapes from gear of the hydraulic motor if the micro-switches fail.

The special metal coating 2.1 and 2.2 is made according to the details A - F. The main stresses on the coating occur on the inlet nozzle at the moment of loading of the reactor and on the cylindrical body, due to the movement of the turning oscillation system. The

stresses are low, and the life time of the coating longer than ten years is determined by the fatigue failure. The sealings are made with an elastomer ring on the discharge cover 2.3, in the bipartition 2.4, between the coating and the reactor shell 2.5. The protection against the melting of the steel and the contamination of the refractory metal weld with Fe is done with sacrifice rings of refractory metal 2.6.

The connections and valves of the reactor are intended for the entrance of water and acidic solution 3.1, entrance of steam 3.2, exit of steam 3.3, vacuum in the reactor body 3.4.1, in the conic sector 3.4.2, in the cover 3.4.3, discharge of the hydrolysate 3.5, valve for collecting pre-hydrolysate for analysis 3.6. The entrance of vapors is at the lower portion and they are distributed by a perforated tube. The entrance of liquid (water or acidic solution) is at the upper portion. Obviously, inlets for liquids and vapors do not present any problem of clogging. The steam outlet is at the upper portion through a perforated pipe, and the hydrolysate discharge is located at the lower portion at the conic sector, which is protected by perforated plate inside the reactor to avoid clogging. A sporadic steam return is foreseen on the plate and perforated tube for unclogging the steam outlet through a by-pass valve between the steam inlet and outlet, after the flexible tube for entrance of steam 3.7 and 3.8. Alternatively, one may use a rotary scraper for the perforated plates. The connections and valves are connected to the inlet supports of the flexible rotation tubes 4 by rigid pipes.

The flexible rotation tubes 5 are constituted by various units: water and acidic solution inlet 5.1, steam inlet 5.2, steam outlet 5.3, vacuum 5.4 and others. The maximum rotation permitted is $\alpha = \pm 45^\circ$ C, so that no liquid will flow through the steam outlet. The level of the acidic solution is maintained below the water line (figure 2.d), so that no liquid will flow out. In case the steam outlet is below the liquid line, one resorts to electric commands of the steam outlet valve 3.3 for closing it, whenever the liquid limit line is exceeded. In general, these electric commands are unnecessary because the oscillating turn of 45° is sufficient to wet the whole biomass, homogenize the liquid and solid mass, and avoid preferred ways in the hydrolysis. The supports of the outlets of the flexible rotation tubes 6 are placed at the upper portion of the reactor.

The intermediate piping 7 are constituted by the same components of the flexible rotation tubes 7.1 - 7.4 and interconnect the outlets of the flexible rotation tubes to the inlets of the flexible tilting tubes.

The flexible tilting tubes 8 are constituted by the same components as the intermediate piping 8.1 - 8.4 and are designed for enabling one to tilt the reactor with its support

structure for discharging of the cellulignin. The outlet of the flexible tilting tubes are connected to the fixed piping 9.1 - 9.4. The latter connect the flexible tilting tubes to the set of valve pipes 9.5 installed on the control panel located beneath the feeder 16.

5 The vacuum sensors 10 are constituted by four units: reactor body 10.1, conic sector 10.2, reactor cover 10.3, and the piping of the vacuum pump 10.4. The closing of the pertinent valves and the viewing of the vacuum drop enable one to determine a possible leakage in the special metal coating.

10 The pressure gauge 11 is of the diaphragm type, in order to resist acidic mediums, and the temperature gauge 12 is protected by a protection shaft of the same material as the coating.

15 The hydraulic system 13 drives the following units in this order: hydraulic motor of the helical feeder 14.1, motion hydraulic cylinder 1.16, hydraulic cylinder of the closing cover 1.10, hydraulic motor of the oscillating rotary system 1.19, hydraulic tilting cylinder 1.14, hydraulic cylinders for opening the wedges 1.11. Please note that there is no simultaneity of any operation, the higher power prevailing, which is that of the hydraulic motor of the helical feeder 14.1.

20 The feeder is composed of a hydraulic motor 14.1, a semi-axle of the propeller 14.2, feeder propeller 14.3, feeder cradle 14.4, a conical feeder nozzle 14.5, and a coil 14.6. The feeder should fill the reactor in a period of 10 minutes. It is built of microbonded carbon steel, protected with bituminous epoxy paint. The space below the feeder is occupied by the control panel 16 (liquids, steams, meters, electric system, etc.), a hydraulic system 13, a vacuum pump 15, a leakage detector 17, work tools and working devices, etc.

25 The fixed structure of the reactor and the feeder accommodate in a standard cart with a platform located at 960 mm from the ground 18, maximum height of the lower equipment at 4.400 mm and within the road standards. The equipment is transported to biomass courts, where it is raised with hydraulic jacks and supported on beams and pillars at the desired processing height (figure 2.e). Alternatively, it can be operated on the transport cart itself.

30 The complementary pieces of equipment of the reactor are: heat exchanger 19, water pressure pump 20.1 and acidic solution discharge 20.2, water deionizer 21, boiler 22, and support structure 23. The latter is transported on standard carts 18, elevated and supported in the same way as the explained before.

Table 5 – Key of the Reactor and Feeder Parts**A – 1st Structure (1st cart)**

	1	– reactor shell and structure
	1.1	– cylindrical body
5	1.2	– cap
	1.3	– inlet nozzle
	1.4	– inlet cover
	1.4.1	– cover
	1.4.2	– hydraulic cylinders
10	1.5	– conical sector
	1.6	– sliding ring with wedges
	1.7	– discharge cover with support for the wedges
	1.8	– connections
	1.9	– hinge
15	1.10	– articulation and hydraulic cylinder cover opener
	1.11	– hydraulic cylinder for closing/opening of wedges
	1.12	– tilting structure
	1.13	– tilting pivot
	1.14	– tilting hydraulic cylinder
20	1.15	– movement cart
	1.16	– horizontal movement hydraulic cylinder
	1.17	– reactor and feeder fixed structure

- 1.18 – chain of the oscillating rotary system
- 1.19 – hydraulic motor of the oscillating rotary system
- 1.20 – micro switches
- 2 – coating in metals and special alloys
- 5 2.1 – body of the reactor
- 2.2 – flanges
- 2.3 – sealing ring of discharge cover (o-ring)
- 2.4 – sealing ring of bipartition (o-ring)
- 2.5 – vacuum sealing ring between shell and coating
- 10 2.6 – Ti ring for protection against Fe contamination in
the Ti weld
- 3 – connections and valves of water, acidic solution, steam,
vacuum, and pre-hydrolysate
- 3.1 – inlet for water and acidic solution
- 15 3.2 – inlet for steam
- 3.3 – outlet for steam
- 3.4 – vacuum
- 3.4.1 – vacuum in the reactor body
- 3.4.2 – vacuum in the conical sector
- 20 3.4.3 – vacuum in the reactor cover
- 3.4.4 – vacuum in the vacuum pump
- 3.5 – discharge of pre-hydrolysate

- 3.6 – valve for collection of pre-hydrolysate for analysis
- 3.7 – steam return valve in the perforated plate of the
pre-hydrolysate discharge pipe
- 3.8 – steam return valve in the perforated tube of the steam outlet
- 5 4 – – entry support of the flexible rotation tubes
- 5 – – flexible rotation tubes
- 5.1 –water and acidic solution inlet
- 5.2 – steam inlet
- 5.3 – steam outlet
- 10 5.4 – vacuum
- 6 – – support for outlet of flexible tubes
- 7 – – intermediate piping
- 7.1 –water and acidic solution inlet
- 7.2 – steam inlet
- 15 7.3 – steam inlet
- 7.4 – vacuum
- 8 – flexible tilting tubes
- 8.1 – steam and acidic solution inlet
- 8.2 – steam inlet
- 20 8.3 – steam outlet
- 8.4 – vacuum
- 9 – piping and valves for water, acidic solution, steam and vacuum

- 9.1 –water and acidic solution inlet
- 9.2 – steam inlet
- 9.3 – steam outlet
- 9.4 – vacuum
- 5 9.5 – valve piping assembly
- 10 10 – sensors
- 10.1 – in the body of the reactor
- 10.2 – in the conical sector
- 10.3 – in the reactor cover
- 10 10.4 – in the vacuum pump
- 11 – pressure gauge
- 12 – temperature gauge
- 13 – hydraulic systems
- 14 – feeder
- 15 14.1 – hydraulic motor
- 14.2 – propellor half-shaft
- 14.3 – feeder propellor
- 14.4 – cradle of the feeder
- 14.5 – conical feeder nozzle
- 20 14.6 – cap
- 15 – vacuum pump
- 16 – control panel (liquids, vapors, meters, electric systems, etc.)

17 – leakage detector

18 – Cart – Capacity 30 t

B – 2nd Structure (2nd cart – figures not included)

19 – heat exchanger

5 20 – pressure pump (20.1) for water and acidic solution (20.2)

21 – water deionizer

22 – boiler

23 – support structure for items 19 to 22

Processing of Pre-Hydrolysis of Biomass and Resulting Products

10 The pre-hydrolysis may be carried out in any type of biomass, such as wood, and sugar-cane bagasse and straw, vegetable residues, barks, grass, organic part of garbage, etc. The pre-hydrolysis process follows these steps:

1- The biomass in minced form is discharged on the feeding platform, which may be constituted by a conveyer or inclined helical feeder, where it is washed in order to diminish
15 the inorganic content (earth, ashes, etc.).

2- The feeding platform unloads the biomass into the helical feeder, which fills the reactor until the beginning of the compacting, reaching the density of 300 kg/m³ (dry matter), which is three times the density of soft biomass (bagasse, straws, grass, vegetable residues, barks, organic part of garbage). The feeder remains coupled to the reactor during the filling
20 operation, which lasts about 10 minutes. After the reactor has been filled, it is uncoupled from the feeder by a horizontal movement of the movable structure with respect to the fixed structure. After the uncoupling, the inlet nozzle is closed by a valve commanded by hydraulic cylinders.

3- After the reactor has been filled, the degasifying operation begins by passing
25 steam, flooding the reactor with a acidic solution (on the order of 1.7% of the biomass), pre-heated up to 80° C in the heat exchanger and in the liquid/solid ratio - L/S = 2, heating and pressurizing up to 160° C, 0.62 MPa. This activity has a duration of 10 minutes, and the heat of the escape steam is recovered in the heat exchanger by the acidic solution of the next re-

action. In order to carry out this recovery of heat, the heat exchanger is usually sized with twice the volume of acidic solution required for each reaction.

4- After heating and pressurizing, the pre-hydrolysis is carried out for a period of 30 minutes simultaneously with the rotary oscillation of the reactor and purge of steam, in order to maintain the temperature close to $160^{\circ}\text{C} \pm 10^{\circ}\text{C}$, which is fundamental to obtain the characteristic of catalytic combustion of cellulignin, since the lower temperatures do not hydrolyze completely the memicellulose and amorphous cellulose, and higher temperatures cause the transformation of crystalline cellulose into amorphous cellulose, thus making it difficult to grind it into fine particles ($f < 250\mu\text{m}$). It should be noticed the importance of the flexible connections to maintain the temperature during the whole period of hydrolysis and the oscillation of the reactor to renew the limit layer of the liquid saturated with sugar on the surface of the chops or particles of biomass, optimizing the hydrolysis period by about 20 - 30 minutes. The control of the pre-hydrolysis is made by accompanying the sugar content in the pre-hydrolysate by means of the refractometer or any other sugar-measuring apparatus up to the value of 9 Bricks. Longer periods of pre-hydrolysis result in a drop of the sugar content due to the decomposition of the xylose into furfural and furfurylic alcohol. The complete control of temperature, pressure, acid content, oscillation (homogenization) and purity of steam permits complete optimization of the pre-hydrolysis process.

5- After the pre-hydrolysis the operation of discharging the pre-hydrolysate (sugar solution) begins, which is directed to the stationary tank or tank-cart through the heat exchanger, thus recovering its energy in the pre-heating of the acidic solution and in the boiler water. The recovery of the heat of the pre-hydrolysate from the escape steam (heating and purge), and the use of low liquid/solid ratio ($L/S=2$) allows one to reduce the consumption of energy of this invention to values lower than 6% of the energy contained in the original biomass. These values are significantly lower than in any other biomass processing, which in most cases are on the order of 30% ($L/S=12$ ratio). The operation of discharge of the hydrolysate lasts about 10 minutes.

6- After the discharge of the pre-hydrolysate, the sugar-recovering washing is carried out in the proportion $L/S = 1$, aiming at a Brick ≥ 5 . This second fraction of the pre-hydrolysate may or may not be mixed in the first fraction (Brick ≥ 9). In general, the first fraction should be intended for the production of furfural (highest possible concentration of sugar), and the second fraction for the production of alcohol (for example, by fermentation with *engineered echerichia coli*). Note that the second fraction contains

(5x1S)/(9x1.5S+5x1S) = 0.27 = 27% of the sugar contained in the two fractions and cannot be lost. Further, it should be noticed that the two fractions totalize $(0.09x1.5S+0.05x1S)/0.2S = 0.925 S = 92.5\%$ of the sugar contained in the hemicellulose and amorphous cellulose. Due to the high recovery rate, there is no need for complex and expensive utilization of the sugars from the washing water, that is discarded. This invention dispenses the use of expensive screw presses for extracting the pre-hydrolysate and recovering sugars. The sugar-recovery period is on the order of 10 minutes.

7- After the recovery of sugar, the discharge of the cellulignin is effected by opening the large cover and tilting the reactor. The cellulignin may be discharged in mechanical washers (conical, cylindrical, rotary tanks, etc.) or on carts to be washed by percolation (diffusion). The discharge is made in a few minutes and, together with the cleaning of the sealing rings and the recovery of the reactor in a discharging position, it takes less than 10 minutes. The complete cycle lasts about 80 minutes, permitting 16 reactions per day, with a rate of utilization of the machine of 90%.

8- The washing operations may be carried out in any type of washer. Preference is given to washing by percolation (diffusion) on carts or big-bags that present lower consumption of water, maintains the cellulignin protected from impurities and permits unloading on bulk carts. The washing waters meet the requirements of Resolution CONAMA No. 20 of June 18, 1986 (published in the D.O.U of July 30, 1996 (Brazilian Official Gazette) and, in principle, they could be discarded in any water vein. With a view to imparting totally ecological characteristics to the invention, the washing waters are neutralized with lime milk and biologically treated with sterilization ponds.

9- There are two washing options in this invention, namely with normal water and with deionized water. With the first water one obtains normal cellulignin with normal ash content resulting from the inorganic materials present in the biomass and impurities from the environment (mainly earth). The washing with deionized water aims at obtaining clean cellulignin to be used as fuel for gas turbines, the greatest requirement of which is the low content of $K + Na < 5$ ppm. The pre-hydrolysis process is a highly efficient process for reducing biomass ashes, where the inorganic materials react with the H_2SO_4 , resulting in soluble sulfates that are leached from the cellulignin in the pre-hydrolysis and in the washing processes. The use of deionized water increases the washing efficiency significantly, enabling the reduction of $Na + K$ content down to the levels required in gas turbines. The use of deionized water in this invention is feasible and inexpensive, in view of the low $L/S = 2$ ratio. In the conventional

processes with a high consumption of water ($L/S = 12$) one has never considered the use of deionized water and, consequently, the use of cellulignin as a fuel for gas turbines.

The biomass pre-hydrolysis process executed by the Failsafe-Type reactor generates two products (two commodities): cellulignin and pre-hydrolysate.

5 CELLULIGNIN

As regards cellulignin, it should be noted that wood is composed of hemicellulose, cellulose and lignin disposed in microfibrillae. The objective of the pre-hydrolysis executed in the Failsafe-Type reactor is to digest the hemicellulose and amorphous cellulose, leaving the cellulose intact in its crystalline form and the lignin in the globulized form. All these features
10 have been achieved and shown in the microphotograph of figure 3. This microphotograph shows the fractal of the fibers of crystalline cellulose with the globulized cellulignin, where the empty spaces were occupied by the hemicellulose and amorphous cellulose prior to the pre-hydrolysis.

This result has the following characteristics:

15 a) due to the complete control of the process parameters of the Failsafe-Type reactor, the characteristics are uniform throughout the mass of the reactor, enabling the production of "homogeneous commodities".

b) Due to the maintenance of the crystallinity, it is possible to grind the cellulignin into particles smaller than 250 μ m in hammer grinders, pin grinders and the like, with a consumption of energy of 12 kWh/t, which applied to Bond's formula: $W=10 Wi/=\frac{10Wi}{P-F}$ whence
20 W = work in kWh/t, Wi = rate of work of the material, P = mesh size through which 80% of the product (in microns) passes, F = mesh size through which 80% of the feed material passes (in microns) results in $Wi = 15$, which is the same order of most mineral materials (that is to say, crystallines). The cost of the grinding energy is lower than US\$ 1.00/t.

25 c) Due to the high porosity, the cellulignin undergoes a dry by natural gravity to content of 50% of moisture, dispensing with expensive screws presses for this processing step. The final drying is made in rotary dryers, achieving moisture lower than 12% with the same consumption of energy of the drying of cereals (750 MJ/t), illustrated by the examples below:

c.1) drying of cellulignin: consumption of 0.4 kg/h of GLP (42 MJ/kg of calorific power) during 10 hours to dry 224 kg of cellulignin - $(0.4 \times 10 \times 42) / 0.224 = 750 \text{ MJ/t cellulignin}$;

c.2) drying of rice: consumption of 1/3 stere (cubic meter) of wood (396 kg/stere, 10.5 MJ/kg of calorific power) to dry 30 sacks of rice a day, 60 kg/sack) - $(0.33 \times 396 \times 10.5) / 1.8$

5 $\approx 760 \text{ MJ/t rice}$.

The cost of drying using noble fuels (wood - US \$10.00/stere - US\$1.80/t of cellulignin or GLP - US\$ 0.50/kg - US\$ 9.00/t of cellulignin) is always very expensive, and so one should use residual heat at 125°C of the boilers. As a result, the drying and grinding should always be made close to the thermoelectric station or boiler, using residual heat;

10 d) Due to the globulization of lignin, empty spaces are opened for attack of the cellulose by microorganisms of ruminant animals, and the cellulignin may be used as a volume component of animal forage with digestibility of 58% comparable to high quality volume components (wet maize silage - 61%, alfalfa silage - 56%, grass forage - 56%, oat straw - 44%, and much higher than the digestibility of natural grass (35%).

15 e) The greatest application of cellulignin is as fuel for boiler, gas turbine and generation of power by magnetohydrodynamics (MHD). Table 6 gives an example of application of cellulignin as fuel for boiler, as compared with fuel oil. The lower calorific power of cellulignin (20 MJ/kg), compared with that of the fuel oil (41 MJ/kg), is counterbalanced by its cost (US\$ 40.00/t), which is 1/3 lower than that of oil (US\$ 120.00/t). Although twice as much cellulignin is required, it is possible for one merely to replace the fuel oil in any type of boiler, since it is an oxygenated fuel (29% of O₂) and requires insufflation of air with volumes close to that of the fuel oil. This means that any oil or gas boiler is capable of burning cellulignin, generating the same thermal power with adjustments on the order of 15% of the burners and air insufflators. The cellulignin produced with deionized water meets the requirements of clean

20 fuel for burning in gas turbines with content of Na + K < 5 ppm. Although the clean cellulignin contains values < 15 ppm, these values are reduced by the cleaning cyclone. For particles smaller than 200 µm, the burning thereof is of the catalytic type, as shown in figure 4. This characteristic results from two factors, the first one being due to the large exposed surface (2 m²/g measured by BET - Brunaur, Emmett and Tellen or the number of iodine 100), compared to the surface of natural biomass (0.4 m²/g), and the second one being due to its grinding into fine particles (d < 250 µm), bringing about the entry of carburant oxygen and the exit of the monoxide in the pores of the cellulignin, without collision between the molecules, giving way to the catalytic combustion (size of cellulignin particle < average free way of the

carburant and gases from the combustion). All indicates that cellulignin is the only solid catalytic fuel that can be produced on a large scale, its period of combustion (< 20 milliseconds) being on the same order as that of natural gas. Gas turbines also require total particulate content < 200 ppm and particulates bigger than 5 mm < 8 ppm. These characteristics are achieved by means of three techniques: complete combustion due to the catalytic characteristic of cellulignin, cyclone installed between the external combustor and the gas turbine and magnetic separator after the grinding of the cellulignin to remove the magnetic contamination originated from the grinders. Without magnetic separators, one reaches values < 50 ppm of total particulates and using magnetic separation one reaches the requirement of 8 ppm < 5 mm. Due to the catalytic combustion, cellulignin is an excellent fuel for MHD, achieving high electric conductivity in the plasma of combustion at high temperature of 2727° C (3.000 K). Figure 5 shows the electric conductivity on the order of 70 S/m, compared with the values of 15 S/m for purified mineral coal, 12 S/m for oil derived from petroleum and 8 S/m for natural gas. It should be noted that wood has carbon content of 48% and hydrogen content of 6%, giving a ratio of C/H = 8. The pre-hydrolysis raises the carbon content in cellulignin to 66% and lowers the H content to 4.3%, doubling the ratio C/H = 15.

Table 6 – Comparison of Oil and Cellulignin Burning in a Conventional Boiler

Project Data			
Projected pressure		7.4 Mpa	
Maximum continuous steam flow – Gauge		67,000 kg/h	
Maximum steam pressure – Gauge		6.5 Mpa	
Steam temperature		485°C	
Feed water temperature		180°C	
Exhaust system		Balanced	
Fuel Data			
Composition (% by weight)	Oil (APF-AI)	Cellulignin	
		Normal ^(a)	Clean ^(b)
Carbon	87.04	66.20	66.30
Hydrogen	11.12	4.30	4.30
Sulphur	0.04	0.10	< 0.01
Nitrogen	0.30	–	–
Oxygen	1.50	29.30	29.40
Ashes	–	0.10	< 15 (Na+K) ^(c)
Higher calorific power	43.7	20.7	20.7
Lower calorific power	41.2	19.8	19.8
(a) Produced with non-deionized water; (b) Produced with deionized water; (c) Values in ppm.			

Cont. Table 6

Fuel Data		
Fuel	Oil	Cellulignin ^(d)
Maximum continuous evaporation rate (kg/h)	67,000	60,300
Steam pressure gauge (Mpa)	6.5	6.5
Steam temperature (°C)	485	485
Feed water temperature (°C)	180	180
Gas temperature at the pre-heater outlet (°C)	180	200
Room temperature of the air (°C)	25	25
Main fuel flow (kg/h)	4,687	8,770
Auxiliary fuel flow of (kg/h)	—	186 ^(e)
Air flow (kg/h)	73,800	84,710
Gas flow (kg/h)	84,040	100,000
Excess air in the combustion (%)	20	13/30 ^(f)
Efficiency (Base PCI) (%)	90.7	86.5
<i>(d) Normal; (e) Natural gas or diesel oil; (f) 13% for cellulignin, 30% for the support</i>		

Table 7 – main inorganic impurities of the eucalyptus and pre-hydrolysate

mg/g	Ca	K	Na	Mg	P	Al	Si	Mn	Fe	Zn	S
Eucalyptus	700	250	—	150	100	50	30	10	8	3	70
Clean cellulignin	1	15	—	80	2	40	100	2	8	—	80
Pre-hydrolysate	280	420	80	160	90	15	30	25	40	5	2,500

5 This result is highly significant, since figure 5 shows that the electric conductivity is very low for low values of C/H, and increases significantly up to C/H = 15, saturating from now onwards. During the combustion, the hydrogen forms the hydroxyl OH⁺, which has a shock section of 400 Å for the electron in the combustion plasma, decreasing its electric conductivity and failing to influence its value to content lower than 3% of H.

10 f) In addition to the applications as fuel and volume component for animal forage, cellulignin has various applications in the following areas: pyrolysis for the production of oils and activated coal, production of carbon black (incomplete combustion), production of methanol, cellulignin resins (agglomerates, MDF - Medium Density Fiber), substrate for semi-solid fermentation (fungi, bacteria and enzymes), etc.

15 PRE-HYDROLYSATE

The pre-hydrolysate is a solution of sugars containing xylose (80%), glucose, mannose, galactose, arabinose, acetyls, uranic acids, etc. In addition to the sugars, it con-

5 tains sulfuric acid added in the pre-hydrolysis process and inorganic impurities given in Table
7. The high control of process of the Failsafe-Type reactor allows one to carry out a virtually
perfect pre-hydrolysis, permitting the digestion of all the products that are easily hydrolyzable,
while preventing the decomposition of the sugars due to the low temperature of the process
10 (160° C). The content of soluble sugars (Bricks) is followed minute by minute, and the process
is discontinued (beginning of the discharge, depressurization, cooling) as soon as the Bricks
passes by a maximum. The low L/S ratio = 2 allows one to achieve sugars content on the
order of 10 Bricks similar to other broths (cane, maize, etc.). The pre-hydrolysate in general
has pH = 1. The recovery of sugars (type of washing with L/S = 1) generates a pre-
10 hydrolysate with Bricks on the order of 5 and pH = 1.5, which may be mixed with the former or
not, for further processing.

The pre-hydrolysate has three main applications: production of furfural by the
two-state technique (dehydration of pentoses), production of ethanol (*engineered echerichia*
coli bacterium) and production of xylitol (catalytic hydrogenation of D. xylose).

15 It should be understood that the example of reactor described together with the
values mentioned are only illustrative and does not limit the protection scope of the invention.

Claims

1. An apparatus for pre-hydrolysis of biomass comprising:

- an external coating (1),

5 - an internal coating (2), the internal coating (2) covering the inside of the external coating (1);

 - there being a space between the internal coating (2) and the external coating (1), said apparatus being characterized in that vacuum (3.4) is produced in the space between the internal coating (2) and the external coating (1).

10 2. An apparatus according to claim 1, characterized in that it includes an oscillation mechanism.

 3. An apparatus according to claim 2, characterized in that the oscillation mechanism enables the apparatus to oscillate around its horizontally arranged axis.

 4. An apparatus according to claim 3, characterized in that the oscillation mechanism permits oscillation in an alternate way at equal intervals.

15 5. An apparatus according to claim 1, characterized in that the vacuum produced is below 1mm of Hg.

 6. An apparatus according to claim 1, characterized in that it is a reactor.

 7. An apparatus according to any one of claims 1 - 6, characterized in that the external coating (1) and the internal coating (2) are made of corrosion resistant material.

20 8. An apparatus according to any one of claims 1, 6 or 7, characterized in that its external coating (1) and internal coating (2) are made of microbonded carbon steel and refractory material, respectively.

 9. An apparatus according to any one of claims 1, 6, 7 or 8, characterized in that the external coating (1) and the internal coating (2) are mounted by simple juxtaposition, free
25 from welding.

10. An apparatus according to any one of claims 1 - 9, characterized by comprising devices for detecting and monitoring the vacuum between the external coating (1) and the internal coating (2).

5 11. An apparatus according to claim 10, characterized in that the detection and monitoring devices detect vacuum microleakage, so as to enable one to detect microcracks in the internal coating (2).

12. An apparatus according to any one of claims 10 or 11, characterized in that the detection of microcracks is made in a continuous way.

10 13. An apparatus according to any one of claims 10 - 12, characterized in that helium gas is introduced into the reactor for detection of microcracks in the internal coating.

14. An apparatus according to any one of claims 7 - 10, characterized in that the microleakage detectors are coupled to the vacuum pump (15).

15. A biomass pre-hydrolysis process comprising the following steps:

- discharging of the biomass into the helical feeder;

15 - starting, in the biomass pre-hydrolysis device, the operation of pressurization, which comprises the following steps:

b.1) flooding the biomass pre-hydrolysis device with a pre-heated acidic solution;
b.2) heating; and b.3) pressurizing;

20 c) said process being characterized in that the pre-hydrolysis is carried out simultaneously with the rotary oscillation of the biomass pre-hydrolysis apparatus, purging of the steam and controlling of temperature, pressure, acid content, time of pre-hydrolysis, and liquid/solid ratio;

d) accompanying the sugar content up to a value close to 10 bricks by means of a sugar meter;

25 e) discharging the pre-hydrolysate in the tank through the heat exchanger;

f) sugar-recovery washing; and

g) discharging the cellulignin into mechanical washers or carts to be washed by percolation.

16. A process according to claim 15, characterized in that the filling of the biomass pre-hydrolysis device is carried out by compacting the biomass in the pre-hydrolysis device.

17. A process according to claims 15 or 16, characterized in that the purging of steam from the pre-hydrolysis device maintains the temperature around 150° C - 170° C.

18. A process according to any one of claims 15, 16, or 17, characterized in that the purging of steam from the pre-hydrolysis device maintains the temperature preferably around 106° C.

19. A process according to any one of claims 15 - 18, characterized in that the pre-hydrolysis is effected during about 30 minutes simultaneously with oscillation and purging of steam.

20. A process according to claim 15, characterized in that the feeding of biomass is carried out on the feeding platform, constituted by an inclined helical conveyor.

21. A process according to claim 15, characterized in that the filling of the biomass pre-hydrolysis device is carried out by the helical feeder as far as the start of compaction, so as to achieve a density around 300 kg/m³.

22. A process according to claim 15, characterized in that the flooding of the biomass pre-hydrolysis device is made with an acidic solution corresponding to 1.7% of the biomass, is pre-heated up to approximately 80° C in the heat exchanger and in the liquid/solid ratio = 2 with heating between 150° C and 170°C, preferably 160°C and pressurization of about 0.62 MPa.

23. A process according to claim 15, characterized in that the heat of the escape steam in the degasing is recovered in the heat exchanger by the acidic solution of the following reaction.

24. A process according to claim 15, characterized by the use of filtered water in the process for producing normal cellulignin and deionized water in the process for producing clean cellulignin.

25. A process according to claim 15, characterized in that the washing waters meet the specifications of fresh waters and may be discarded in any water stream.

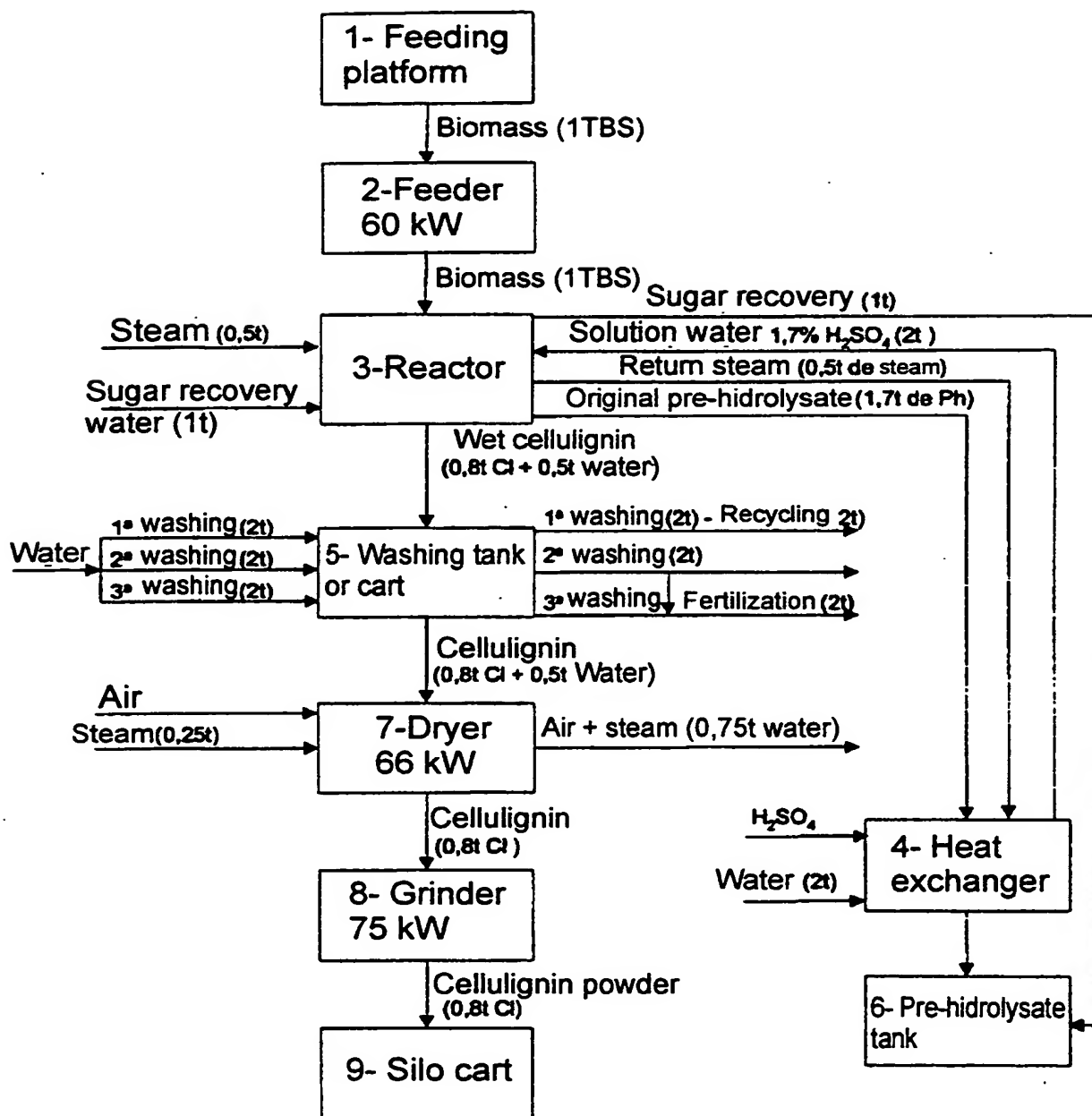
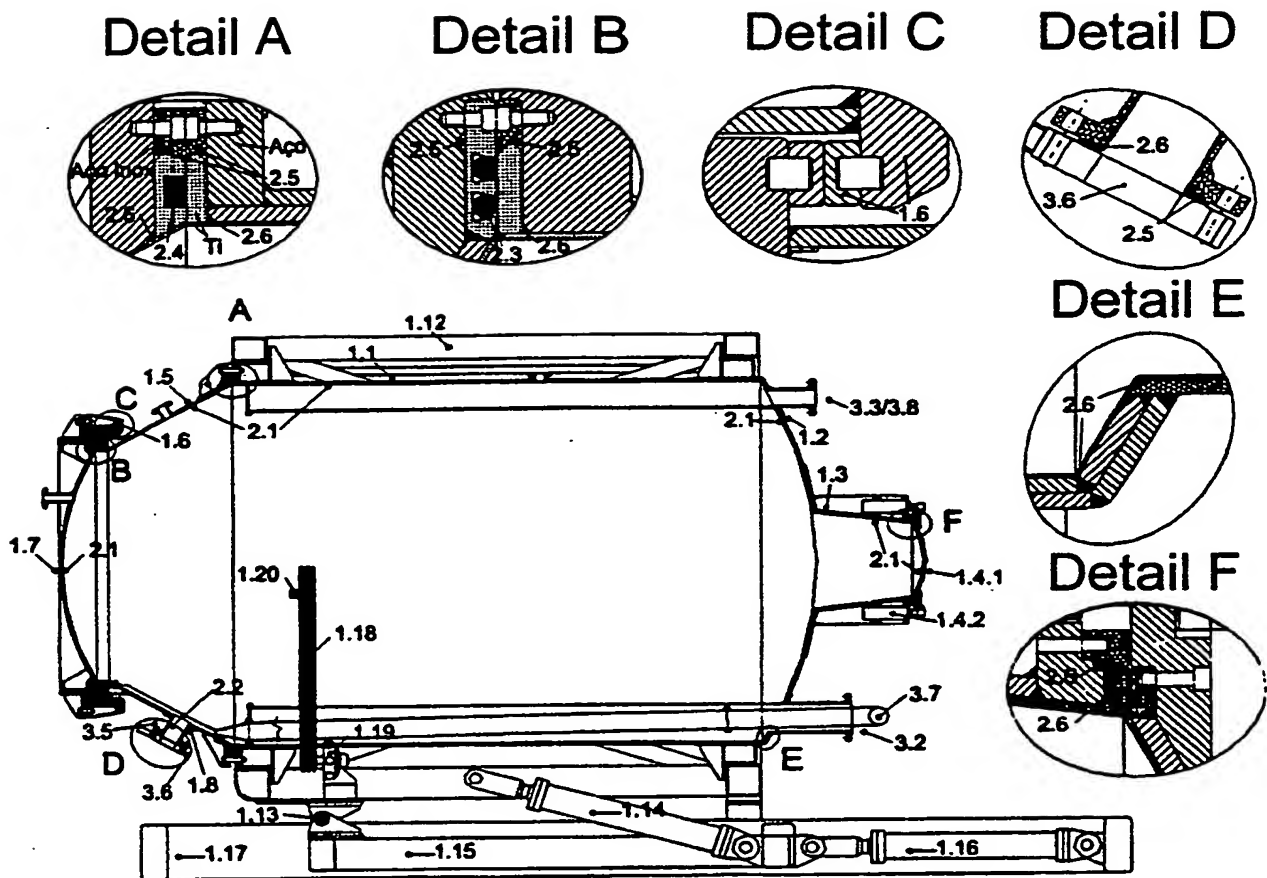
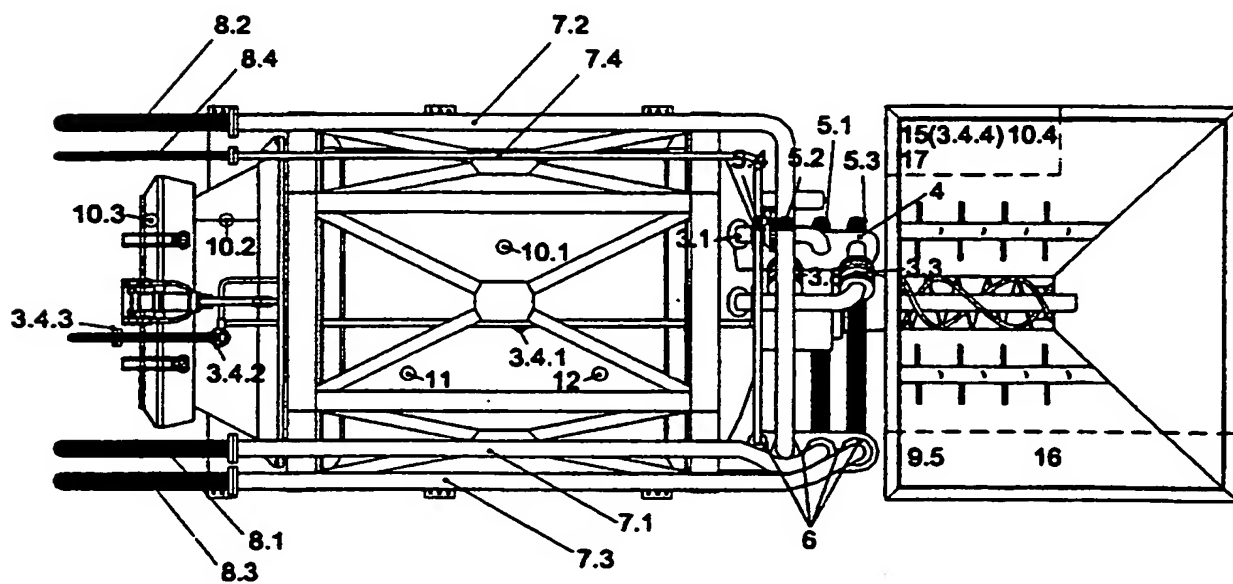
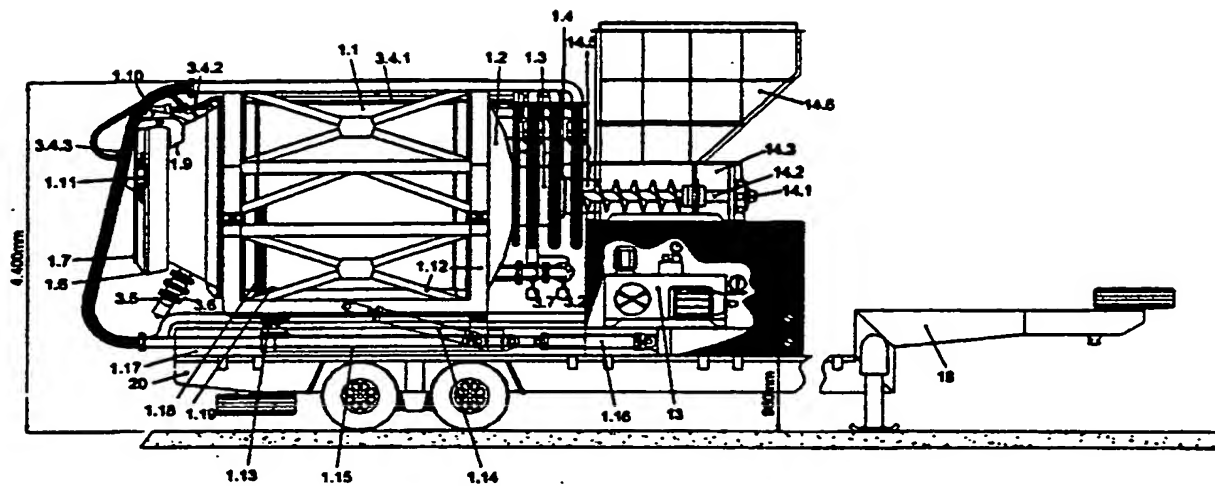


FIG. 1

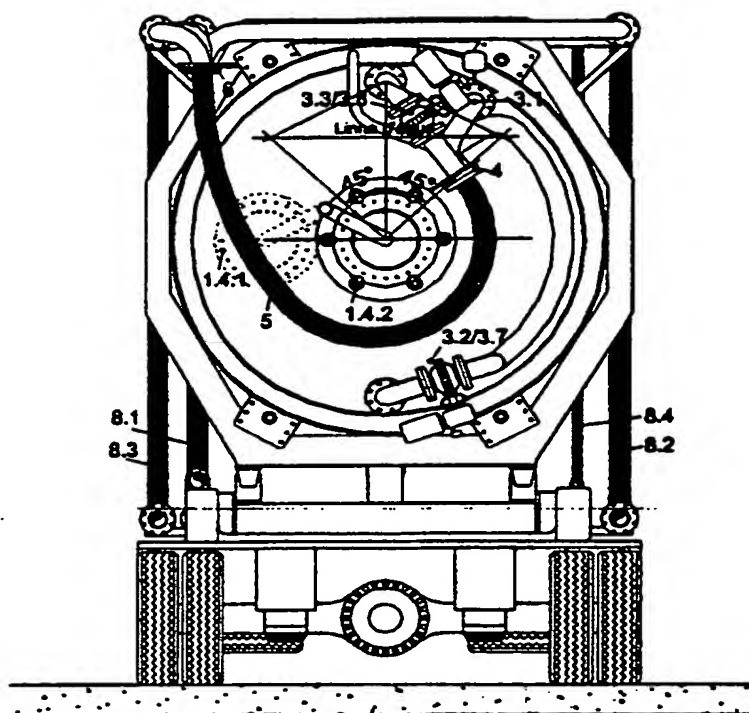
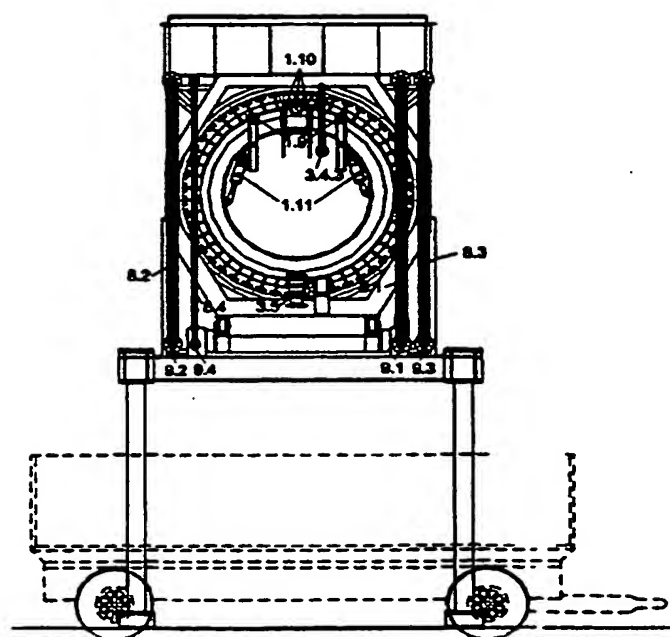
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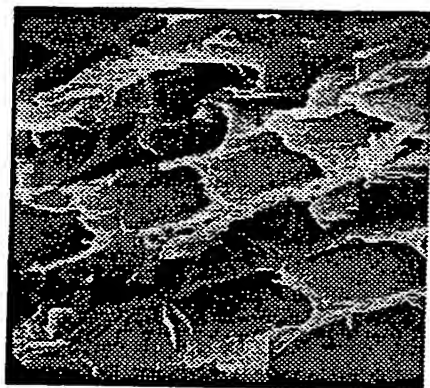
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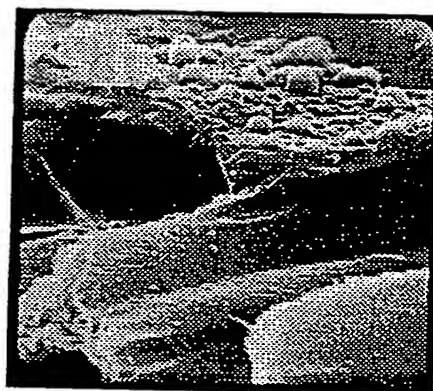
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**FIG. 2d****FIG. 2**

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1800 X, 1 mm = 370 nm



5000 X, 1 mm = 133 nm

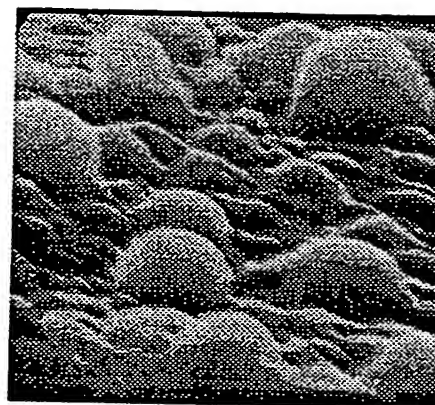
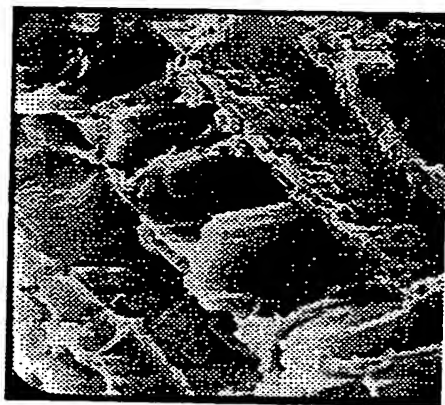
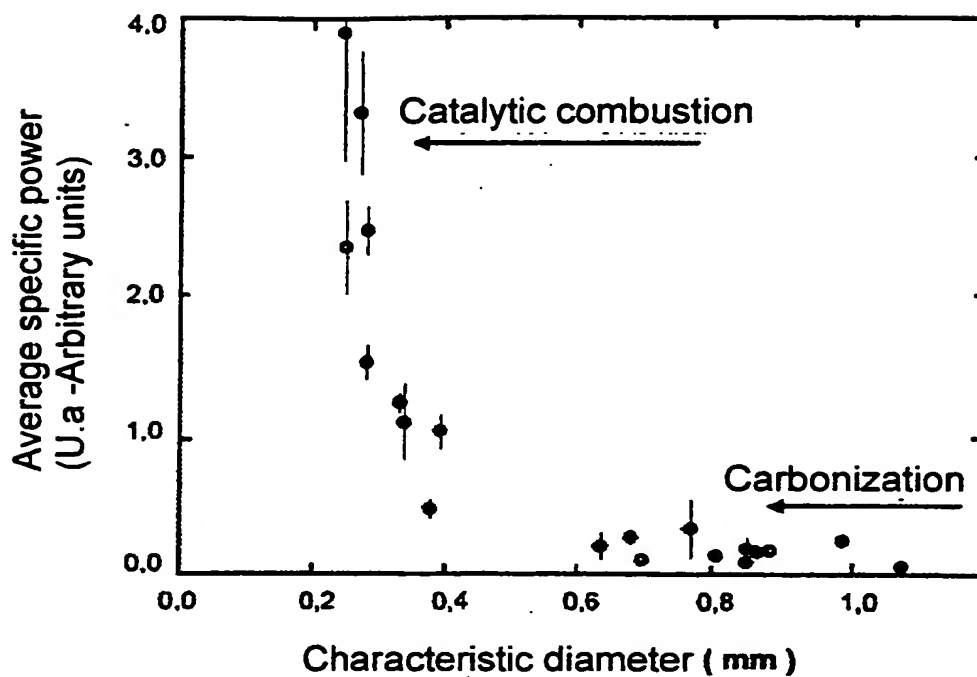
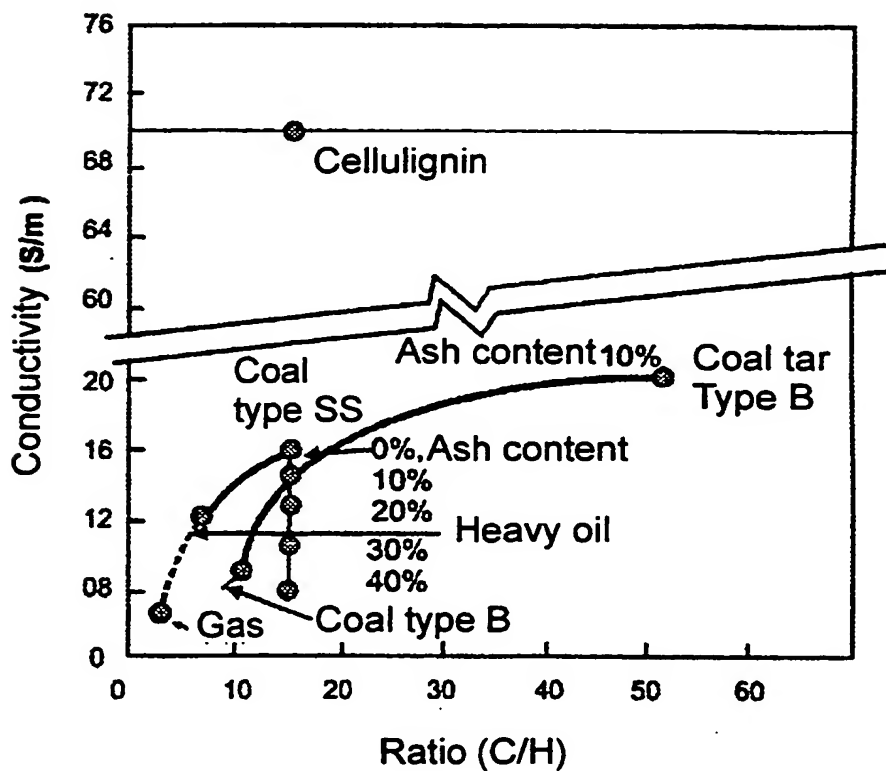


FIG. 3

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**FIG. 4****FIG. 5**

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Rio, August 30, 2001

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Ref.: PCT - International patent application no. PCT/BR00/00065
filed on June 23, 2000
RM MATERIAIS REFRATÁRIOS LTDA.
Our ref.: PE-3849 (sst)

Dear Sirs:

With reference to the Invitation to Restrict or to Pay Additional Fees, mailed on July 30 2001, we herein provide the following comments:

1) The applicant has chosen to pay the extra fee of EUR 1,533.00 in order to continue the examination. The remittance of EUR 1533.00 has been made to your account no. 33388000 at the Dresdner Bank (see attached copy of the corresponding transaction record).

2) As mentioned in item 3, sheet 1 of the annex, the applicant will present its arguments to differentiate the prior art documents, classified as "X", from its own invention. After the applicant's comments it will be verified that no amendments are necessary to be made in the claims to overcome the cited references;

2.1 Comments on the present invention:

1. In Patent application PCT/BR00/00065, the inventive concept is a single pressure vessel constituted of two juxtaposed materials that are maintained in position by a vacuum between them.

2. The material of the external wall has a structural function that resists the vessel's internal pressure, and the internal lining material resists the corrosion inherent in the pre-hydrolysis and hydrolysis process.

3. The vacuum in this invention has the function of maintaining the materials juxtaposed in order to prevent eventual implosion of the internal lining, in case the internal vessel pressure becomes negative due to the condensation of vapors entailed in the process.

4. Each material is chosen and dimensioned to perform a specific function:

4.1 The external wall material is made of a micro-alloy carbon steel and its thickness is designed to provide the vessel with mechanical strength. Due to the high mechanical strength of carbon steel, the thickness of this layer is low.

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4.2 The internal lining is a refractory material (Ti, Zr, Nb, Ta and their alloys) presenting high resistance to corrosion and abrasion, which is, therefore, an extremely fine layer.

5. The vacuum established between the external wall material and the lining material, coupled with the employment of leak detection techniques, allows monitoring of the occurrence of micro-cracks in the internal lining material (typical among refractory materials). This type of detection is very accurate, capable of detecting even those microscopic fissures that do not affect the reactor's operation. This is the concept of the **reactor failure of the saf typ** where, with proper monitoring, one can work with materials that present welding crack problems, intrinsic to the materials utilized. Thus, the reactor can be operated normally without any harm to its performance or durability.

6. The technology of **juxtaposition of two materials** and the exploitation of the best properties of each material allows the construction of chemical reactors, without limitations regarding different constructive shapes and dimensions appropriate for the processing of solids (biomass and minerals) in **corrosive environments**, particularly in the acid pre-hydrolysis process.

7. The technology of **juxtaposition of two materials** results in a fine-wall pressure vessel (concept of an eggshell). The reactor is, therefore, a **pi ce of light equipment** that can be constructed on the chassis of a lorry so as to provide full mobility. It also allows the provision of a mechanism for oscillation of the vessel, providing homogenization of the product and less reaction time. Moreover, it provides a mechanism for tilting the reactor and the construction of reactors with large flanges / covers suitable for the unloading of solid materials.

Comments on Patent US 3,056,664

1. In the apparatus described in Patent US 3,056,664, **the vacuum is contained in a separate vessel**, called jacket (10), detached from the main vessel with pressure containment (2). See Figure 1.

2. The container (16), being made of "Pyrex" or "Vycor", is resistant to corrosion. It is a vessel independent from the vacuum vessel, which, in turn, is independent from the pressure vessel. See Specification column 2, lines 40-50, and column 3, line 44-56.

3. The vacuum in this invention has the purpose of thermal insulation. See Specification column 3, lines 52-56

Conclusion: The inventive concepts described in our invention and in Patent US 3,056,664 are **completely different**: In our invention, there is a **single pr ssure vessel constituted of two juxtaposed materials**. The vacuum is maintained between the two juxtaposed materials and has the basic function of preventing implosion of the internal lining material in case the pressure in the reactor becomes negative. The inventive concept of Patent US 3,056,664 is the **thr indep ndent v ss ls**, on insid the other. The vacuum is maintained in the separate intermediate vessel and has the sole function of thermal insulation.

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Comments on Patent WO 00/31459

1. It is described in Patent Application WO 00/31459 that the vacuum is established between two structurally independent vessels (vessels 2 and 3), which are concentric and spatially separated from one another.
2. The vacuum in this invention has the purpose of thermal insulation. This invention is aimed at construction of a means for stocking and transport of cryogenic fluids.
3. Besides the vacuum, this Patent provides multi-layered insulation (cryogenic super thermal insulation consisting of aluminium sheets or films that reflect radiation, intercalated with sheets of insulating material).

Conclusion: The inventive concept described in our invention and in Patent WO 00/31459 is completely different: In our invention, there is a single pressure vessel constituted of two juxtaposed materials. The vacuum is maintained between two juxtaposed materials and has the basic function of preventing the implosion of the internal lining material in case the pressure in the reactor becomes negative. The inventive concept of Patent Application WO 00/31459 is the construction of two independent vessels, one inside the other. The vacuum maintained between them has the function of thermal insulation. The technology described in our invention has nothing to do with cryogenic fluids and besides, Patent application PCT/BR00/0065 refers to a reactor.

Comments on Patent US 4,997,124

1. In the apparatus (vacuum bottle or frostfree pipe – see Specification column 1, lines 9 & 10) described in Patent US 4,997,124, the vacuum is established between two structurally independent vessels (vessels 2 and 3), which are concentric and spatially separated from each other.
2. The vacuum in this invention has the purpose of thermal insulation.
3. The Patent refers to the substitution in vacuum bottles of the deposition of radiation reflecting layers by coating with sheets of activated copper, titanium and zirconium. These activated metals are intended to reflect the radiation and absorb gases that may remain in the space or leave the surface of the inner and outer shells defining the vacuum space. See Specification column 2 & 3, lines 66-5.

Conclusion: The inventive concept described in our invention and in Patent US 4,997,124 is again completely different: In our invention, there is a single pressure vessel constituted of two juxtaposed materials. The vacuum is maintained between two juxtaposed materials and has the basic function of preventing the implosion of the internal lining material in case the pressure in the reactor becomes negative. The inventive concept of Patent US 4,997,124 is two independent vessels, one inside the other. The vacuum maintained between them has the function of thermal insulation of the vacuum bottle.

In view of the comments provided above, the applicant believes that the novelty and inventive step of the invention are clear.

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With regard to Annex sheet 1, item 4, obviously, the applicant would like to receive the international preliminary examination report (IPER) in good time so as to be able to enter the national phases of its choice. Nevertheless, only if the examiner is unable to complete his examination within the established period, would we prefer to receive the IPER later, provided that no damage in the regular prosecution shall be sustained.

Very truly yours,

Dannemann, Siemsen, Bigler & Ipanema Moreira

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PATENT COOPERATION TREATY

T2SD.

From the
INTERNATIONAL PRELIMINARY EXAMINATION AUTHORITY

PCT

INVITATION TO RESTRICT OR
TO PAY ADDITIONAL FEES

(PCT Article 34(3) (a) and Rule 68.2)

T :

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Date of mailing (Day/month/year)	30.07.2001
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Applicant's or agent's file reference
PE-3849

REPLY OR PAYMENT DUE	within 1 month(s) from the above date of mailing
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International application No.
PCT/BR00/00065

International filing date (day/month/year)
23/06/2000

Priority date (day/month/year)
23/06/1999

International Patent classification (IPC) or national Patent classification:
B01J19/02

Applicant

RM MATERIAIS REFRATARIOS LTDA. et al.

1. This International Examining Authority

- (i) considers that the international application does not comply with the requirements of unity of invention (Rule 13.1, 13.2 and 13.3) for the reasons indicated in the Annex.
- (ii) therefore considers that there are 2 inventions claimed in the international application as indicated in the Annex.
- (iii) recalls that claims relating to inventions in respect of which no international search report has been established need not be the subject of international preliminary examination (Rule 66.1 (e)).

2. Consequently the applicant is hereby invited, within the time limit indicated above, to restrict the claims as suggested under item 3, below, or to pay the amount indicated below:

EUR 1533.00	x	001	=	EUR 1533.00
Fee per additional invention		number of additional inventions		total amount of additional fees

The applicant is informed that, according to Rule 68.3 (c), the payment of any additional fee may be made under protest, i.e. a reasoned statement to the effect that the international application complies with the requirement of unity of invention or that the amount of the required additional fee is excessive.

- 3. If the applicant opts to restrict the claims, this Authority suggests the restriction possibilities indicated in the Annex, which in its opinion would be in compliance with the requirement of unity of invention.
- 4. In the absence of any response from the applicant, this Authority will establish the international preliminary examination report on those parts of the international application indicated in the Annex which, in the opinion of this Authority appear to relate to the main invention.

Name and mailing address of the
international preliminary examination authority:

European Patent Office
D-80298 Munich
Tel. +49 89 2399 - 0 Tx: 523656 pmu d
Fax: +49 89 2399 - 4465

Authorized officer

Buesing, G

Telephone No. +49 89 2399-8356



André

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**INVITATION TO RESTRICT
OR TO PAY ADDITIONAL FEES**

International application No. PCT/BR00/00065

1. An objection concerning lack of unity of invention has already been raised by the International Search Authority (ISA). It is noted that the applicant has paid an additional search fee for the second invention.
2. The International Preliminary Examination Authority (IPEA) concurs with the objection and the reasons therefore as raised by the ISA and indicated in its communication dated 07.11.2000.

In the absence of any response, the international preliminary examination will be carried out for the first invention as identified in the International Search Report.

3. It is noted that the examiner entrusted with the examination of this application received this file only recently, and it is observed that there is little time left for carrying out the international preliminary examination within the time limit defined under the PCT.

Taking account of the international search report, it appears that prior art documents relevant for novelty and/or inventive step of the claimed subject-matter has been cited. In order to expedite the examination, ~~the applicant is invited to comment the relevance of the cited prior art documents and to amend the claims as appropriate.~~ This applies in particular to the documents classified as "X" in the search report.

4. In view of the short period left for terminating the preliminary international examination, ~~the applicant is invited to indicate in its response whether it is considered acceptable to receive the international preliminary examination report (IPEP) later than required by the PCT or whether a possibly negative IPEP may be issued directly, i.e. without a further communication.~~

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PATENT COOPERATION TREATY

inv. SST
9FN
23.07.01
PCT

From the INTERNATIONAL SEARCHING AUTHORITY

To:-
**DANNEMANN, SIEMSEN, BIGLER &
 IPANEMA MOREIRA**
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 BRAZIL

NOTIFICATION OF TRANSMITTAL OF
 THE INTERNATIONAL SEARCH REPORT
 OR THE DECLARATION

(PCT Rule 44.1)

19
 15
 14
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DANNEMANN, SIEMSEN, BIGLER & IPANEMA MOREIRA

Applicant's or agent's file reference PE-3849	Date of mailing (day/month/year) 12/07/2001
International application No. PCT/BR 00/ 00065	International filing date (day/month/year) 23/06/2000
Applicant RM MATERIAIS REFRATARIOS LTDA.	

1. ☒ The applicant is hereby notified that the International Search Report has been established and is transmitted herewith.

Filing of amendments and statement under Article 19:
 The applicant is entitled, if he so wishes, to amend the claims of the International Application (see Rule 46):

When? The time limit for filing such amendments is normally 2 months from the date of transmittal of the International Search Report; however, for more details, see the notes on the accompanying sheet.

Where? Directly to the International Bureau of WIPO
 34, chemin des Colombettes
 1211 Geneva 20, Switzerland
 Facsimile No.: (41-22) 740.14.35

For more detailed instructions, see the notes on the accompanying sheet.

2. ☐ The applicant is hereby notified that no International Search Report will be established and that the declaration under Article 17(2)(a) to that effect is transmitted herewith.

3. ☐ With regard to the protest against payment of (an) additional fee(s) under Rule 40.2, the applicant is notified that:

☐ the protest together with the decision thereon has been transmitted to the International Bureau together with the applicant's request to forward the texts of both the protest and the decision thereon to the designated Offices.
☐ no decision has been made yet on the protest; the applicant will be notified as soon as a decision is made.

4. **Further action(s):** The applicant is reminded of the following:

Shortly after 18 months from the priority date, the international application will be published by the International Bureau. If the applicant wishes to avoid or postpone publication, a notice of withdrawal of the international application, or of the priority claim, must reach the International Bureau as provided in Rules 90bis.1 and 90bis.3, respectively, before the completion of the technical preparations for international publication.

Within 19 months from the priority date, a demand for international preliminary examination must be filed if the applicant wishes to postpone the entry into the national phase until 30 months from the priority date (in some Offices even later).

Within 20 months from the priority date, the applicant must perform the prescribed acts for entry into the national phase before all designated Offices which have not been elected in the demand or in a later election within 19 months from the priority date or could not be elected because they are not bound by Chapter II.

Name and mailing address of the International Searching Authority European Patent Office, P.B. 5818 Patentlaan 2 NL-2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Authorized officer Emmanuel Cherqui
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NOTES TO FORM PCT/ISA/220

These Notes are intended to give the basic instructions concerning the filing of amendments under article 19. The Notes are based on the requirements of the Patent Cooperation Treaty, the Regulations and the Administrative Instructions under that Treaty. In case of discrepancy between these Notes and those requirements, the latter are applicable. For more detailed information, see also the PCT Applicant's Guide, a publication of WIPO.

In these Notes, "Article", "Rule", and "Section" refer to the provisions of the PCT, the PCT Regulations and the PCT Administrative Instructions, respectively.

INSTRUCTIONS CONCERNING AMENDMENTS UNDER ARTICLE 19

The applicant has, after having received the international search report, one opportunity to amend the claims of the international application. It should however be emphasized that, since all parts of the international application (claims, description and drawings) may be amended during the international preliminary examination procedure, there is usually no need to file amendments of the claims under Article 19 except where, e.g. the applicant wants the latter to be published for the purposes of provisional protection or has another reason for amending the claims before international publication. Furthermore, it should be emphasized that provisional protection is available in some States only.

What parts of the international application may be amended?

Under Article 19, only the claims may be amended.

During the international phase, the claims may also be amended (or further amended) under Article 34 before the International Preliminary Examining Authority. The description and drawings may only be amended under Article 34 before the International Examining Authority.

Upon entry into the national phase, all parts of the international application may be amended under Article 28 or, where applicable, Article 41.

When?

Within 2 months from the date of transmittal of the international search report or 16 months from the priority date, whichever time limit expires later. It should be noted, however, that the amendments will be considered as having been received on time if they are received by the International Bureau after the expiration of the applicable time limit but before the completion of the technical preparations for international publication (Rule 46.1).

Where not to file the amendments?

The amendments may only be filed with the International Bureau and not with the receiving Office or the International Searching Authority (Rule 46.2).

Where a demand for international preliminary examination has been/is filed, see below.

How?

Either by cancelling one or more entire claims, by adding one or more new claims or by amending the text of one or more of the claims as filed.

A replacement sheet must be submitted for each sheet of the claims which, on account of an amendment or amendments, differs from the sheet originally filed.

All the claims appearing on a replacement sheet must be numbered in Arabic numerals. Where a claim is cancelled, no renumbering of the other claims is required. In all cases where claims are renumbered, they must be renumbered consecutively (Administrative Instructions, Section 205(b)).

The amendments must be made in the language in which the international application is to be published.

What documents must/may accompany the amendments?

Letter (Section 205(b)):

The amendments must be submitted with a letter.

The letter will not be published with the international application and the amended claims. It should not be confused with the "Statement under Article 19(1)" (see below, under "Statement under Article 19(1)").

The letter must be in English or French, at the choice of the applicant. However, if the language of the international application is English, the letter must be in English; if the language of the international application is French, the letter must be in French.

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NOTES TO FORM PCT/ISA/220 (continued)

The letter must indicate the differences between the claims as filed and the claims as amended. It must, in particular, indicate, in connection with each claim appearing in the international application (it being understood that identical indications concerning several claims may be grouped), whether

- (i) the claim is unchanged;
- (ii) the claim is cancelled;
- (iii) the claim is new;
- (iv) the claim replaces one or more claims as filed;
- (v) the claim is the result of the division of a claim as filed.

The following examples illustrate the manner in which amendments must be explained in the accompanying letter:

1. [Where originally there were 48 claims and after amendment of some claims there are 51]:
"Claims 1 to 29, 31, 32, 34, 35, 37 to 48 replaced by amended claims bearing the same numbers; claims 30, 33 and 36 unchanged; new claims 49 to 51 added."
2. [Where originally there were 15 claims and after amendment of all claims there are 11]:
"Claims 1 to 15 replaced by amended claims 1 to 11."
3. [Where originally there were 14 claims and the amendments consist in cancelling some claims and in adding new claims]:
"Claims 1 to 6 and 14 unchanged; claims 7 to 13 cancelled; new claims 15, 16 and 17 added." or
"Claims 7 to 13 cancelled; new claims 15, 16 and 17 added; all other claims unchanged."
4. [Where various kinds of amendments are made]:
"Claims 1-10 unchanged; claims 11 to 13, 18 and 19 cancelled; claims 14, 15 and 16 replaced by amended claim 14; claim 17 subdivided into amended claims 15, 16 and 17; new claims 20 and 21 added."

"Statement under article 19(1)" (Rule 46.4)

The amendments may be accompanied by a statement explaining the amendments and indicating any impact that such amendments might have on the description and the drawings (which cannot be amended under Article 19(1)).

The statement will be published with the international application and the amended claims.

It must be in the language in which the international application is to be published.

It must be brief, not exceeding 500 words if in English or if translated into English.

It should not be confused with and does not replace the letter indicating the differences between the claims as filed and as amended. It must be filed on a separate sheet and must be identified as such by a heading, preferably by using the words "Statement under Article 19(1)."

It may not contain any disparaging comments on the international search report or the relevance of citations contained in that report. Reference to citations, relevant to a given claim, contained in the international search report may be made only in connection with an amendment of that claim.

Consequence if a demand for International preliminary examination has already been filed

If, at the time of filing any amendments and any accompanying statement, under Article 19, a demand for international preliminary examination has already been submitted, the applicant must preferably, at the time of filing the amendments (and any statement) with the International Bureau, also file with the International Preliminary Examining Authority a copy of such amendments (and of any statement) and, where required, a translation of such amendments for the procedure before that Authority (see Rules 55.3(a) and 62.2, first sentence). For further information, see the Notes to the demand form (PCT/IPEA/401).

Consequence with regard to translation of the international application for entry into the national phase

The applicant's attention is drawn to the fact that, upon entry into the national phase, a translation of the claims as amended under Article 19 may have to be furnished to the designated/elected Offices, instead of, or in addition to, the translation of the claims as filed.

For further details on the requirements of each designated/elected Office, see Volume II of the PCT Applicant's Guide.

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PATENT COOPERATION TREATY

PCT

INTERNATIONAL SEARCH REPORT

(PCT Article 18 and Rules 43 and 44)

Applicant's or agent's file reference PE-3849	FOR FURTHER ACTION <small>see Notification of Transmittal of International Search Report (Form PCT/ISA/220) as well as, where applicable, item 5 below.</small>	
International application No. PCT/BR 00/ 00065	International filing date (day/month/year) 23/06/2000	(Earliest) Priority Date (day/month/year) 23/06/1999
Applicant RM MATERIAIS REFRATARIOS LTDA.		

This International Search Report has been prepared by this International Searching Authority and is transmitted to the applicant according to Article 18. A copy is being transmitted to the International Bureau.

This International Search Report consists of a total of 5 sheets.
☒ It is also accompanied by a copy of each prior art document cited in this report.

1. Basis of the report

- a. With regard to the language, the international search was carried out on the basis of the international application in the language in which it was filed, unless otherwise indicated under this item.
- ☐ the international search was carried out on the basis of a translation of the international application furnished to this Authority (Rule 23.1(b)).
- b. With regard to any nucleotide and/or amino acid sequence disclosed in the international application, the international search was carried out on the basis of the sequence listing :
- ☐ contained in the international application in written form.
- ☐ filed together with the international application in computer readable form.
- ☐ furnished subsequently to this Authority in written form.
- ☐ furnished subsequently to this Authority in computer readable form.
- ☐ the statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.
- ☐ the statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished

2. ☐ Certain claims were found unsearchable (See Box I).

3. ☒ Unity of invention is lacking (see Box II).

4. With regard to the title,

- ☒ the text is approved as submitted by the applicant.
- ☐ the text has been established by this Authority to read as follows:

5. With regard to the abstract,

- ☒ the text is approved as submitted by the applicant.
- ☐ the text has been established, according to Rule 38.2(b), by this Authority as it appears in Box III. The applicant may, within one month from the date of mailing of this international search report, submit comments to this Authority.

6. The figure of the drawings to be published with the abstract is Figure No.

- ☒ as suggested by the applicant.
- ☐ because the applicant failed to suggest a figure.
- ☐ because this figure better characterizes the invention.
- 2a
☐ None of the figures.

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INTERNATIONAL SEARCH REPORT

National Application No

PCT/BR 00/00065

A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 B01J19/02 B01J19/28 C13K1/02

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 B01J C13K F16L F17C

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, WPI Data, PAJ

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 3 056 664 A (DRAVNIKS ANDREWS, FOREST PARK, TROSCINSKI EDWIN S, BIRKNESS HARALD A) 2 October 1962 (1962-10-02) the whole document	1,6,7,9
P,X	WO 00 31459 A (STEYR DAIMLER PUCH AG ;BRUNNHOFER KLAUS (AT)) 2 June 2000 (2000-06-02) the whole document	1,5,9
X	US 4 997 124 A (KITABATAKE AKIHIRO ET AL) 5 March 1991 (1991-03-05) column 5, line 9 - line 21 column 6, line 22 - line 42 column 8, line 56 -column 9, line 20 figures 1,2,9,10	1,5

-/-



Further documents are listed in the continuation of box C.



Patent family members are listed in annex.

* Special categories of cited documents :

A document defining the general state of the art which is not considered to be of particular relevance

E earlier document but published on or after the international filing date

L document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

O document referring to an oral disclosure, use, exhibition or other means

P document published prior to the international filing date but later than the priority date claimed

T later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

X document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

Y document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

Z document member of the same patent family

Date of the actual completion of the international search

5 July 2001

Date of mailing of the international search report

12 07. 2001

Name and mailing address of the ISA

European Patent Office, P.B. 5818 Patentlaan 2
NL - 2280 HV Rijswijk
Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,
Fax (+31-70) 340-3016

Authorized officer

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INTERNATIONAL SEARCH REPORT

International Application No.

P/BR 00/00065

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	✓ WO 99 26720 A (ALLIED SIGNAL INC) 3 June 1999 (1999-06-03) page 5, line 1 -page 6, line 11 page 7, line 9 - line 21 page 8, line 12 -page 9, line 14 claims 1-16; figure 1	1,6,7, 9-14
A	✓ US 5 159 694 A (OVERATH HORST, SOEDER CARL-JOHANNES, SALHANI NAZIR) 27 October 1992 (1992-10-27) column 5, line 65 -column 6, line 12 figure 4	1-4,6, 15-25
A	✓ GB 1 569 138 A (VALKANAS ET AL.) 11 June 1980 (1980-06-11) claims; example 1	15-25
A	✓ US 4 199 371 A (REGNAULT ET AL.) 22 April 1980 (1980-04-22) claims; examples	15-25

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INTERNATIONAL SEARCH REPORT

International application No.
PCT/BR 00/00065

Box I Observations where certain claims were found unsearchable (Continuation of item 1 of first sheet)

This International Search Report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. ☐ Claims Nos.:
because they relate to subject matter not required to be searched by this Authority, namely:
2. ☐ Claims Nos.:
because they relate to parts of the International Application that do not comply with the prescribed requirements to such an extent that no meaningful International Search can be carried out, specifically:
3. ☐ Claims Nos.:
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).

Box II Observations where unity of invention is lacking (Continuation of item 2 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

see additional sheet

1. ☒ As all required additional search fees were timely paid by the applicant, this International Search Report covers all searchable claims.
2. ☐ As all searchable claims could be searched without effort justifying an additional fee, this Authority did not invite payment of any additional fee.
3. ☐ As only some of the required additional search fees were timely paid by the applicant, this International Search Report covers only those claims for which fees were paid, specifically claims Nos.:
4. ☐ No required additional search fees were timely paid by the applicant. Consequently, this International Search Report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:

Remark on Protest

- ☐ The additional search fees were accompanied by the applicant's protest.
- ☒ No protest accompanied the payment of additional search fees.

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FURTHER INFORMATION CONTINUED FROM PCT/SA/ 210

This International Searching Authority found multiple (groups of) inventions in this international application, as follows:

1. Claims: 1-14

Independent claim 1 discloses an apparatus comprising an external and an internal coating and a space between the two coatings adapted to be under vacuum. The dependent claims disclose further details of the apparatus concerning an oscillation mechanism, the pressure within said space, the materials and the way of mounting of the coatings and a leakage detection system.

2. Claims: 15-25

Independent claim 15 discloses a process of biomass pre-hydrolysis characterized in that said process is carried out with a rotary oscillation apparatus. The dependent claims disclose further details of said process.

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INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

P R 00/00065

Patent document cited in search report		Publication date	Patent family member(s)	Publication date
US 3056664	A	02-10-1962	NONE	
WO 0031459	A	02-06-2000	AT 3144 U	25-10-1999
US 4997124	A	05-03-1991	JP 1268521 A	26-10-1989
			JP 1970865 C	18-09-1995
			JP 6098108 B	07-12-1994
			JP 1297022 A	30-11-1989
			JP 6098109 B	07-12-1994
			KR 9209830 B	31-10-1992
WO 9926720	A	03-06-1999	AU 1601099 A	15-06-1999
US 5159694	A	27-10-1992	DE 3608466 A	17-09-1987
			DE 3632093 A	24-03-1988
			AT 77830 T	15-07-1992
			DE 3780044 A	06-08-1992
			DE 3780044 D	06-08-1992
			EP 0237039 A	16-09-1987
			ES 2033707 T	01-04-1993
			JP 62244384 A	24-10-1987
GB 1569138	A	11-06-1980	AR 221819 A	31-03-1981
			AU 504082 B	04-10-1979
			AU 1881076 A	27-04-1978
			BR 7607104 A	13-09-1977
			DE 2647910 A	05-05-1977
			EG 12428 A	30-06-1979
			ES 452669 A	01-04-1978
			FR 2328795 A	20-05-1977
			IN 146710 A	18-08-1979
			IN 145513 A	28-10-1978
			IT 1076827 B	27-04-1985
			JP 52081103 A	07-07-1977
			PT 65746 A, B	01-11-1976
			ZA 7606073 A	28-09-1977
US 4199371	A	22-04-1980	CH 609092 A	15-02-1979
			AT 361418 B	10-03-1981
			AT 220578 A	15-07-1980
			AU 518576 B	08-10-1981
			AU 3466478 A	04-10-1979
			BE 865584 A	02-10-1978
			BR 7802044 A	19-12-1978
			CA 1100492 A	05-05-1981
			CU 34898 A	10-01-1981
			DE 2814067 A	12-10-1978
			DK 144578 A	02-10-1978
			EG 13177 A	31-12-1980
			ES 468437 A	01-01-1979
			FI 780956 A, B,	02-10-1978
			FR 2395314 A	19-01-1979
			GB 1562682 A	12-03-1980
			IT 1093515 B	19-07-1985
			JP 53124632 A	31-10-1978
			MX 5047 E	02-03-1983
			NL 7803360 A	03-10-1978
			NO 781128 A, B,	03-10-1978

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INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

BR 00/00065

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 4199371 A		NZ 186826 A	19-06-1979
		OA 5924 A	30-06-1981
		PL 205735 A	15-01-1979
		SE 439648 B	24-06-1985
		SE 7803578 A	02-10-1978
		US 4257818 A	24-03-1981
		US 4304608 A	08-12-1981

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PATENT COOPERATION TREATY

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non agenda
11-12-800
18-10-800
22-12-900

PCT

From the INTERNATIONAL SEARCHING AUTHORITY

To:
DANNEMANN, SIEMSEN, BIGLER &
IPANEMA MOREIRA
Rua Marques de Olinda 70, Botafogo
Caixa Postal 2142
22251-040- Rio de Janeiro - RJ
BRAZIL

INVITATION TO PAY ADDITIONAL FEES

(PCT Article 17(3)(a) and Rule 40.1)

Applicant's or agent's file reference PE-3849	Date of mailing (day/month/year) 07/11/2000
International application No. PCT/BR 00/ 00065	PAYMENT DUE within 45 days/days from the above date of mailing
International filing date (day/month/year) 23/06/2000	
Applicant RM MATERIAIS REFRATARIOS LTDA.	

1. This International Searching Authority

- (i) considers that there are 2 (number of) inventions claimed in the international application covered by the claims indicated ~~on~~ on the extra sheet:

and it considers that the international application does not comply with the requirements of unity of invention (Rules 13.1, 13.2 and 13.3) for the reasons indicated ~~on~~ on the extra sheet:

- (ii) ☒ has carried out a partial international search (see Annex) ☐ will establish the international search report on those parts of the international application which relate to the invention first mentioned in claims Nos.:

1-14

- (iii) will establish the international search report on the other parts of the international application only if, and to the extent to which, additional fees are paid

2. The applicant is hereby invited, within the time limit indicated above, to pay the amount indicated below:

DEM 1.848,26 x 1 = DEM 1.848,26
 Fee per additional invention number of additional inventions total amount of additional fees

Or, EUR 945,00 x 1 = EUR 945,00

The applicant is informed that, according to Rule 40.2(c), the payment of any additional fee may be made under protest, i.e., a reasoned statement to the effect that the international application complies with the requirement of unity of invention or that the amount of the required additional fee is excessive.

3. ☐ Claim(s) Nos. _____ have been found to be unsearchable under Article 17(2)(b) because of defects under Article 17(2)(a) and therefore have not been included with any invention.

Name and mailing address of the International Searching Authority



European Patent Office, P.B. 5818 Patentlaan 2
 NL-2280 HV Rijswijk
 Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,
 Fax: (+31-70) 340-3016

Authorized officer

Toñi Muñoz-Manneken

André

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This International Searching Authority found multiple (groups of) inventions in this international application, as follows:

1. Claims: 1-14

Independent claim 1 discloses an apparatus comprising an external and an internal coating and a space between the two coatings adapted to be under vacuum. The dependent claims disclose further details of the apparatus concerning an oscillation mechanism, the pressure within said space, the materials and the way of mounting of the coatings and a leakage detection system.

2. Claims: 15-25

Independent claim 15 discloses a process of biomass pre-hydrolysis characterized in that said process is carried out with a rotary oscillation apparatus. The dependent claims disclose further details of said process.

Specification according to Rule 40.1 PCT of the reasons for which the International Application PCT/BR00/00065 filed on 23 June 2000 is not considered as complying with the requirements of unity of invention according to Rule 13 PCT.

According to the Administrative Instructions under the PCT (in force from 1 July 1998), Annex B, Part 1(c), unity of invention has to be considered in the first place only in relation to the independent claims.

The application contains two independent claims: claim 1 (apparatus claim) and claim 15 (process claim).

Independent claim 1 discloses an apparatus comprising an external and an internal coating and a space between the two coatings adapted to operate under vacuum.

Independent claim 15 discloses a process of biomass pre-hydrolysis characterized in that said process is carried out with a rotary oscillation apparatus.

The only common (same or corresponding) technical features among those two independent claims is an apparatus. This is a priori not novel but well known and fundamental in the art.

Thus, there are no common special (novel and inventive) technical features present between independent claims 1 and 15 as required by Rule 13.2 PCT.

The only common problem among the two above mentioned independent claims which could be recognized is the provision of an apparatus suitable for treating biomass. This problem is a priori very well known in the art.

Thus, there is no common problem among the independent claims 1 and 15, serving as a single common general inventive concept required by Rule

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13.1 PCT.

Consequently, two groups of alleged inventions could be discovered in the application:

Group I (independent claim 1 and dependent claims 2-14) related to an apparatus comprising two coatings having a space between them adapted to operate under vacuum.

Group II (independent claim 15 and dependent claims 16-25) related to a process for biomass pre-hydrolysis characterized in that said process is carried out in a rotary oscillation apparatus.

According to Article 17(3)(a) PCT the ISA shall establish the International Search Report on those parts of the International Application which relate to the invention first mentioned in the claims, i.e. the above mentioned Group I.

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1. The present communication is an Annex to the invitation to pay additional fees (Form PCT/ISA/206). It shows the results of the international search established on the parts of the international application which relate to the invention first mentioned in claims Nos.:

- 1-14
2. This communication is not the international search report which will be established according to Article 18 and Rule 43.
3. If the applicant does not pay any additional search fees, the information appearing in this communication will be considered as the result of the international search and will be included as such in the international search report.
4. If the applicant pays additional fees, the international search report will contain both the information appearing in this communication and the results of the international search on other parts of the international application for which such fees will have been paid.

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 3 056 664 A (DRAVNIKS ANDREWS, FOREST PARK, TROSCINSKI EDWIN S, BIRKNESS HARALD A) 2 October 1962 (1962-10-02) the whole document	1, 6, 7, 9
P, X	WO 00 31459 A (STEYR DAIMLER PUCH AG ; BRUNNHOFER KLAUS (AT)) 2 June 2000 (2000-06-02) the whole document	1, 5, 9
X	US 4 997 124 A (KITABATAKE AKIHIRO ET AL) 5 March 1991 (1991-03-05) column 5, line 9 - line 21 column 6, line 22 - line 42 column 8, line 56 - column 9, line 20 figures 1, 2, 9, 10	1, 5
A	WO 99 26720 A (ALLIED SIGNAL INC) 3 June 1999 (1999-06-03) page 5, line 1 - page 6, line 11 page 7, line 9 - line 21 page 8, line 12 - page 9, line 14 claims 1-16; figure 1	1, 6, 7, 9-14
A	US 5 159 694 A (OVERATH HORST, SOEDER CARL-JOHANNES, SALHANI NAZIR) 27 October 1992 (1992-10-27) column 5, line 65 - column 6, line 12 figure 4	1-4, 6

☐ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

* Special categories of cited documents :

- *A* document defining the general state of the art which is not considered to be of particular relevance
- *E* earlier document but published on or after the international filing date
- *L* document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- *O* document referring to an oral disclosure, use, exhibition or other means
- *P* document published prior to the international filing date but later than the priority date claimed

- *T* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
- *X* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
- *Y* document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
- *Z* document member of the same patent family

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Patent Family Annex

Information on patent family members

International Application No

PCT 00/00065

Patent document cited in search report		Publication date	Patent family member(s)		Publication date
US 3056664	A	02-10-1962	NONE		
WO 0031459	A	02-06-2000	AT	3144 U	25-10-1999
US 4997124	A	05-03-1991	JP	1268521 A	26-10-1989
			JP	1970865 C	18-09-1995
			JP	6098108 B	07-12-1994
			JP	1297022 A	30-11-1989
			JP	6098109 B	07-12-1994
			KR	9209830 B	31-10-1992
WO 9926720	A	03-06-1999	AU	1601099 A	15-06-1999
US 5159694	A	27-10-1992	DE	3608466 A	17-09-1987
			DE	3632093 A	24-03-1988
			AT	77830 T	15-07-1992
			DE	3780044 A	06-08-1992
			DE	3780044 D	06-08-1992
			EP	0237039 A	16-09-1987
			ES	2033707 T	01-04-1993
			JP	62244384 A	24-10-1987

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The demand must be filed directly with the competent International Preliminary Examining Authority or two or more Authorities are competent, with the one chosen by the applicant. The full name or two-letter code of that Authority may be indicated by the applicant on the line below:

IPEA/ EPO

PCT

CHAPTER II

DEMAND

under Article 31 of the Patent Cooperation Treaty:
The undersigned requests that the international application specified below be the subject of international preliminary examination according to the Patent Cooperation Treaty and hereby elects all eligible States (except where otherwise indicated).

For International Preliminary Examining Authority use only	
Identification of IPEA	Date of receipt of DEMAND
Box No. I IDENTIFICATION OF THE INTERNATIONAL APPLICATION Applicant's or agent's file reference PE-3849	
International application No. PCT/BR00/00065	International filing date (day/month/year) 23 June 2000 (23.06.2000)
(Earliest) Priority date (day/month/year) 23 June 1999 (23.06.99)	
Title of invention "An apparatus and process for pre-hydrolysis of biomass"	
Box No. II APPLICANT(S)	
Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.) RM MATERIAIS REFRATÁRIOS LTDA. Estrada do Pinhal, 750 12600-000 - Lorena - SP Brazil	Telephone No.: Facsimile No.: Teleprinter No.:
State (that is, country) of nationality: BR	State (that is, country) of residence: BR
Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.) GARCIA PINATTI, DALTRO Rua Oswaldo Aranha, 1194 - casa 1, Vila Zelia Lorena - SP Brazil	
State (that is, country) of nationality: BR	State (that is, country) of residence: BR
Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.) GUEDES SOARES, ALVARO Rua Madre Amanda de Castro Junqueira, 117 Mirante Mogi Mirim - São Paulo Brazil	
State (that is, country) of nationality: BR	State (that is, country) of residence: BR
<input type="checkbox"/> Further applicants are indicated on a continuation sheet.	

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Box N . III AGENT OR COMMON REPRESENTATIVE; OR ADDRESS FOR CORRESPONDENCEThe following person is ☒ agent ☐ common representativeand ☒ has been appointed earlier and represents the applicant(s) also for international preliminary examination.☐ is hereby appointed and any earlier appointment of (an) agent(s)/common representative is hereby revoked.☐ is hereby appointed, specifically for the procedure before the International Preliminary Examining Authority, in addition to the agent(s)/common representative appointed earlier.Name and address: *(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.)*DANNEMANN, SIEMSEN, BIGLER & IPANEMA MOREIRA
Caixa Postal 2142
Rua Marquês de Olinda, 70
Botafogo
22251-040 - Rio de Janeiro - RJ
Brazil

Telephone No.:

(5521) 553.1811

Facsimile No.:

(5521) 553.1812
553.1813

Teleprinter No.:

☐ Address for correspondence: Mark this check-box where no agent or common representative is/has been appointed and the space above is used instead to indicate a special address to which correspondence should be sent.**Box No. IV BASIS FOR INTERNATIONAL PRELIMINARY EXAMINATION****Statement concerning amendments:***

1. The applicant wishes the international preliminary examination to start on the basis of:

☒ the international application as originally filedthe description ☒ as originally filed☐ as amended under Article 34the claims ☒ as originally filed☐ as amended under Article 19 (together with any accompanying statement)☐ as amended under Article 34the drawings ☒ as originally filed☐ as amended under Article 342. ☐ The applicant wishes any amendment to the claims under Article 19 to be considered as reversed.3. ☐ The applicant wishes the start of the international preliminary examination to be postponed until the expiration of 20 months from the priority date unless the International Preliminary Examining Authority receives a copy of any amendments made under Article 19 or a notice from the applicant that he does not wish to make such amendments (Rule 69.1(d)). *(This check-box may be marked only where the time limit under Article 19 has not yet expired.)*

* Where no check-box is marked, international preliminary examination will start on the basis of the international application as originally filed or, where a copy of amendments to the claims under Article 19 and/or amendments of the international application under Article 34 are received by the International Preliminary Examining Authority before it has begun to draw up a written opinion or the international preliminary examination report, as so amended.

Language for the purposes of international preliminary examination: English☒ which is the language in which the international application was filed.☐ which is the language of a translation furnished for the purposes of international search.☐ which is the language of publication of the international application.☐ which is the language of the translation (to be) furnished for the purposes of international preliminary examination.**Box No. V ELECTION OF STATES**The applicant hereby elects all eligible States *(that is, all States which have been designated and which are bound by Chapter II of the PCT)*

excluding the following States which the applicant wishes not to elect:

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Box No. VI CHECK LIST

The demand is accompanied by the following elements, in the language referred to in Box No. IV, for the purposes of international preliminary examination:

- | | | |
|--|---|--------|
| 1. translation of international application | : | sheets |
| 2. amendments under Article 34 | : | sheets |
| 3. copy (or, where required, translation) of amendments under Article 19 | : | sheets |
| 4. copy (or, where required, translation) of statement under Article 19 | : | sheets |
| 5. letter | : | sheets |
| 6. other (specify) | : | sheets |

For International Preliminary Examining Authority use only

received not received


<input type="checkbox"/>	<input type="checkbox"/>
<input type="checkbox"/>	<input type="checkbox"/>
<input type="checkbox"/>	<input type="checkbox"/>
<input type="checkbox"/>	<input type="checkbox"/>
<input type="checkbox"/>	<input type="checkbox"/>
<input type="checkbox"/>	<input type="checkbox"/>

The demand is also accompanied by the item(s) marked below:

- | | |
|--|---|
| 1. <input checked="" type="checkbox"/> fee calculation sheet | 4. <input type="checkbox"/> statement explaining lack of signature |
| 2. <input type="checkbox"/> separate signed power of attorney | 5. <input type="checkbox"/> nucleotide and or amino acid sequence listing in computer readable form |
| 3. <input type="checkbox"/> copy of general power of attorney; reference number, if any: | 6. <input type="checkbox"/> other (specify): |

Box No. VII SIGNATURE OF APPLICANT, AGENT OR COMMON REPRESENTATIVE

Next to each signature, indicate the name of the person signing and the capacity in which the person signs (if such capacity is not obvious from reading the demand).


Dannemann, Siemsen, Bigler & Ipanema Moreira

For International Preliminary Examining Authority use only

- Date of actual receipt of DEMAND:
- Adjusted date of receipt of demand due to CORRECTIONS under Rule 60.1(b):
- ☐ The date of receipt of the demand is AFTER the expiration of 19 months from the priority date and item 4 or 5, below, does not apply.
 ☐ The applicant has been informed accordingly.
- ☐ The date of receipt of the demand is WITHIN the period of 19 months from the priority date as extended by virtue of Rule 80.5.
- ☐ Although the date of receipt of the demand is after the expiration of 19 months from the priority date, the delay in arrival is EXCUSED pursuant to Rule 82.

For International Bureau use only

Demand received from IPEA on:

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PCT

FEE CALCULATION SHEET

Annex to the Demand for international preliminary examination

International application No. PCT/BR00/00065	For International Preliminary Examining Authority use only	
Applicant's or agent's file reference PE-3849	Date stamp of the IPEA	
Applicant RM MATERIAIS REFRATÁRIOS LTDA.		
Calculation of prescribed fees		
1. Preliminary examination fee	DEM 749,58	<input type="checkbox"/> P
2. Handling fee <i>(Applicants from certain States are entitled to a reduction of 75% of the handling fee. Where the applicant is (or all applicants are) so entitled, the amount to be entered at H is 25% of the handling fee.)</i>	DEM 287,51	<input type="checkbox"/> H
3. Total of prescribed fees Add the amounts entered at P and H and enter total in the TOTAL box	DEM 1.037,09	
TOTAL		
Mode of Payment		
<input type="checkbox"/> authorization to charge deposit account with the IPEA (see below)	<input type="checkbox"/> cash	
<input type="checkbox"/> cheque	<input type="checkbox"/> revenue stamps	
<input type="checkbox"/> postal money order	<input type="checkbox"/> coupons	
<input checked="" type="checkbox"/> bank draft	<input type="checkbox"/> other (specify):	
Deposit Account Authorization <i>(this mode of payment may not be available at all IPEAs)</i>		
The IPEA/ _____ <input type="checkbox"/> is hereby authorized to charge the total fees indicated above to my deposit account.		
<input type="checkbox"/> <i>(this check-box may be marked only if the conditions for deposit accounts of the IPEA so permit)</i> is hereby authorized to charge any deficiency or credit any overpayment in the total fees indicated above to my deposit account.		
Deposit Account Number _____	Date (day/month/year) _____	Signature _____

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TPC

From the
INTERNATIONAL PRELIMINARY EXAMINATION AUTHORITY

To:

DANNEMANN, SIEMSEN, BIGLER &
IPANEMA MOREIRA
Rua Marquês de Olinda 70
Caixa Postal 2142
Botafogo
22251-040- Rio de Janeiro - RJ
BRESIL

PCT

INVITATION TO RESTRICT OR
TO PAY ADDITIONAL FEES

(PCT Article 34(3) (a) and Rule 68.2)

Applicant's or agent's file reference PE-3849		Date of mailing (Day/month/year) 30.07.2001
International application No. PCT/BR00/00065		REPLY OR PAYMENT DUE within 1 month(s) from the above date of mailing
International filing date (day/month/year) 23/06/2000	Priority date (day/month/year) 23/06/1999	
International Patent classification (IPC) or national Patent classification: B01J19/02		
Applicant RM MATERIAIS REFRATARIOS LTDA. et al.		

1. This International Examining Authority

- (i) considers that the international application does not comply with the requirements of unity of invention (Rule 13.1, 13.2 and 13.3) for the reasons indicated in the Annex.
- (ii) therefore considers that there are 2 inventions claimed in the international application as indicated in the Annex.
- (iii) recalls that claims relating to inventions in respect of which no international search report has been established need not be the subject of international preliminary examination (Rule 66.1 (e)).

2. Consequently the applicant is hereby invited, within the time limit indicated above, to restrict the claims as suggested under item 3, below, or to pay the amount indicated below:

$$\frac{\text{EUR 1533.00}}{\text{Fee per additional invention}} \times \frac{001}{\text{number of additional inventions}} = \frac{\text{EUR 1533.00}}{\text{total amount of additional fees}}$$

The applicant is informed that, according to Rule 68.3 (c), the payment of any additional fee may be made under protest, i.e. a reasoned statement to the effect that the international application complies with the requirement of unity of invention or that the amount of the required additional fee is excessive.

- 3. If the applicant opts to restrict the claims, this Authority suggests the restriction possibilities indicated in the Annex, which in its opinion would be in compliance with the requirement of unity of invention.
- 4. In the absence of any response from the applicant, this Authority will establish the international preliminary examination report on those parts of the international application indicated in the Annex which, in the opinion of this Authority appear to relate to the main invention.

Name and mailing address of the
international preliminary examination authority:

European Patent Office
D-80298 Munich
Tel. +49 89 2399 - 0 Tx: 523656 epmu d
Fax: +49 89 2399 - 4465

Authorized officer

Buesing, G

Telephone No. +49 89 2399-8356



Amber

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**INVITATION TO RESTRICT
OR TO PAY ADDITIONAL FEES**

International application No. PCT/BR00/00065

1. An objection concerning lack of unity of invention has already been raised by the International Search Authority (ISA). It is noted that the applicant has paid an additional search fee for the second invention.
2. The International Preliminary Examination Authority (IPEA) concurs with the objection and the reasons therefore as raised by the ISA and indicated in its communication dated 07.11.2000.

In the absence of any response, the international preliminary examination will be carried out for the first invention as identified in the International Search Report.

3. It is noted that the examiner entrusted with the examination of this application received this file only recently, and it is observed that there is little time left for carrying out the international preliminary examination within the time limit defined under the PCT.

Taking account of the international search report, it appears that prior art documents relevant for novelty and/or inventive step of the claimed subject-matter has been cited. In order to expedite the examination, the applicant is invited to comment the relevance of the cited prior art documents and to amend the claims as appropriate. This applies in particular to the documents classified as "X" in the search report.

4. In view of the short period left for terminating the preliminary international examination, the applicant is invited to indicate in its response whether it is considered acceptable to receive the international preliminary examination report (IPER) later than required by the PCT or whether a possibly negative IPER may be issued directly, ie without a further communication.

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PCT

REQUEST

The undersigned requests that the present international application be processed according to the Patent Cooperation Treaty.

For receiving Office use only

International Application No. 00 / 0 0065
International Filing Date 23 JUN 2000 20-8-00
INFORMATIONAL APPLICATION
Name of receiving Office and "PCT International Application"
Applicant's or agent's file reference (if desired) (12 characters maximum) PE-3849

Box No. I TITLE OF INVENTION

"AN APPARATUS AND PROCESS FOR PRE-HYDROLYSIS OF BIOMASS"

Box No. II APPLICANT

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)

RM MATERIAIS REFRAATÓRIOS LTDA.
Estrada do Pinhal, 750
12600-000 - Lorena - SP
Brazil

☐ This person is also inventor.

Telephone No.

Facsimile No.

Teleprinter No.

State (that is, country) of nationality:

BR

State (that is, country) of residence:

BR

This person is applicant for the purposes of:

☐ all designated States

☒ all designated States except the United States of America

☐ the United States of America only

☐ the States indicated in the Supplemental Box

Box No. III FURTHER APPLICANT(S) AND/OR (FURTHER) INVENTOR(S)

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)

GARCIA PINATTI, DALTRO
Rua Oswaldo Aranha, 1194, casa 1
Vila Zelia
Lorena, São Paulo
Brazil

This person is:

☐ applicant only

☒ applicant and inventor

☐ inventor only (If this check-box is marked, do not fill in below.)

State (that is, country) of nationality:

BR

State (that is, country) of residence:

BR

This person is applicant for the purposes of:

☐ all designated States

☐ all designated States except the United States of America

☒ the United States of America only

☐ the States indicated in the Supplemental Box

☒ Further applicants and/or (further) inventors are indicated on a continuation sheet.

Box No. IV AGENT OR COMMON REPRESENTATIVE; OR ADDRESS FOR CORRESPONDENCE

The person identified below is hereby/has been appointed to act on behalf of the applicant(s) before the competent International Authorities as:

☒ agent

☐ common representative

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.)

DANNEMANN, SIEMSEN, BIGLER & IPANEMA MOREIRA
Caixa Postal 2142
Rua Marquês de Olinda, 70
Botafogo
22251-040 - Rio de Janeiro - RJ
Brazil

Telephone No.

(21) 553.1811

Facsimile No.

(21) 553.1812
553.1813

Teleprinter No.

☐ Address for correspondence: Mark this check-box where an agent or common representative is/has been appointed and the space above is used instead to indicate a special address to which correspondence should be sent.

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Continuation of Box No. III FURTHER APPLICANT(S) AND/OR (FURTHER) INVENTOR(S)

If none of the following sub-boxes is used, this sheet should not be included in the request.

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)

GUEDES SOARES, ALVARO
Rua Madre Amanda de Castro Junqueira, 117
Mirante
Mogi Mirim, São Paulo
Brazil

This person is:

- ☐ applicant only
☒ applicant and inventor
☐ inventor only (If this check-box is marked, do not fill in below.)

State (that is, country) of nationality:

BR

State (that is, country) of residence:

BR

This person is applicant for the purposes of:

- ☐ all designated States ☐ all designated States except the United States of America ☒ the United States of America only ☐ the States indicated in the Supplemental Box

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)

This person is:

- ☐ applicant only
☐ applicant and inventor
☐ inventor only (If this check-box is marked, do not fill in below.)

State (that is, country) of nationality:

State (that is, country) of residence:

This person is applicant for the purposes of:

- ☐ all designated States ☐ all designated States except the United States of America ☐ the United States of America only ☐ the States indicated in the Supplemental Box

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)

This person is:

- ☐ applicant only
☐ applicant and inventor
☐ inventor only (If this check-box is marked, do not fill in below.)

State (that is, country) of nationality:

State (that is, country) of residence:

This person is applicant for the purposes of:

- ☐ all designated States ☐ all designated States except the United States of America ☐ the United States of America only ☐ the States indicated in the Supplemental Box

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)

This person is:

- ☐ applicant only
☐ applicant and inventor
☐ inventor only (If this check-box is marked, do not fill in below.)

State (that is, country) of nationality:

State (that is, country) of residence:

This person is applicant for the purposes of:

- ☐ all designated States ☐ all designated States except the United States of America ☐ the United States of America only ☐ the States indicated in the Supplemental Box

☐ Further applicants and/or (further) inventors are indicated in another continuation sheet.

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Box N .V DESIGNATION OF STATES

The following designations are hereby made under Rule 4.9(a) (mark the applicable check-boxes; at least one must be marked):

Regional Patent

- ☒ **AP** ARIPO Patent: GH Ghana, GM Gambia, KE Kenya, LS Lesotho, MW Malawi, SD Sudan, SL Sierra Leone, SZ Swaziland, TZ United Republic of Tanzania, UG Uganda, ZW Zimbabwe, and any other State which is a Contracting State of the Harare Protocol and of the PCT
- ☒ **EA** Eurasian Patent: AM Armenia, AZ Azerbaijan, BY Belarus, KG Kyrgyzstan, KZ Kazakhstan, MD Republic of Moldova, RU Russian Federation, TJ Tajikistan, TM Turkmenistan, and any other State which is a Contracting State of the Eurasian Patent Convention and of the PCT
- ☒ **EP** European Patent: AT Austria, BE Belgium, CH and LI Switzerland and Liechtenstein, CY Cyprus, DE Germany, DK Denmark, ES Spain, FI Finland, FR France, GB United Kingdom, GR Greece, IE Ireland, IT Italy, LU Luxembourg, MC Monaco, NL Netherlands, PT Portugal, SE Sweden, and any other State which is a Contracting State of the European Patent Convention and of the PCT
- ☒ **OA** OAPI Patent: BF Burkina Faso, BJ Benin, CF Central African Republic, CG Congo, CI Côte d'Ivoire, CM Cameroon, GA Gabon, GN Guinea, GW Guinea-Bissau, ML Mali, MR Mauritania, NE Niger, SN Senegal, TD Chad, TG Togo, and any other State which is a member State of OAPI and a Contracting State of the PCT (if other kind of protection or treatment desired, specify on dotted line)

National Patent (if other kind of protection or treatment desired, specify on dotted line):

- | | |
|---|---|
| <input checked="" type="checkbox"/> AE United Arab Emirates | <input checked="" type="checkbox"/> LR Liberia |
| <input checked="" type="checkbox"/> AL Albania | <input checked="" type="checkbox"/> LS Lesotho |
| <input checked="" type="checkbox"/> AM Armenia | <input checked="" type="checkbox"/> LT Lithuania |
| <input checked="" type="checkbox"/> AT Austria | <input checked="" type="checkbox"/> LU Luxembourg |
| <input checked="" type="checkbox"/> AU Australia | <input checked="" type="checkbox"/> LV Latvia |
| <input checked="" type="checkbox"/> AZ Azerbaijan | <input checked="" type="checkbox"/> MA Morocco |
| <input checked="" type="checkbox"/> BA Bosnia and Herzegovina | <input checked="" type="checkbox"/> MD Republic of Moldova |
| <input checked="" type="checkbox"/> BB Barbados | <input checked="" type="checkbox"/> MG Madagascar |
| <input checked="" type="checkbox"/> BG Bulgaria | <input checked="" type="checkbox"/> MK The former Yugoslav Republic of Macedonia |
| <input checked="" type="checkbox"/> BR Brazil | <input checked="" type="checkbox"/> MN Mongolia |
| <input checked="" type="checkbox"/> BY Belarus | <input checked="" type="checkbox"/> MW Malawi |
| <input checked="" type="checkbox"/> CA Canada | <input checked="" type="checkbox"/> MX Mexico |
| <input checked="" type="checkbox"/> CH and LI Switzerland and Liechtenstein | <input checked="" type="checkbox"/> NO Norway |
| <input checked="" type="checkbox"/> CN China | <input checked="" type="checkbox"/> NZ New Zealand |
| <input checked="" type="checkbox"/> CR Costa Rica | <input checked="" type="checkbox"/> PL Poland |
| <input checked="" type="checkbox"/> CU Cuba | <input checked="" type="checkbox"/> PT Portugal |
| <input checked="" type="checkbox"/> CZ Czech Republic | <input checked="" type="checkbox"/> RO Romania |
| <input checked="" type="checkbox"/> DE Germany | <input checked="" type="checkbox"/> RU Russian Federation |
| <input checked="" type="checkbox"/> DK Denmark | <input checked="" type="checkbox"/> SD Sudan |
| <input checked="" type="checkbox"/> DM Dominica | <input checked="" type="checkbox"/> SE Sweden |
| <input checked="" type="checkbox"/> EE Estonia | <input checked="" type="checkbox"/> SG Singapore |
| <input checked="" type="checkbox"/> ES Spain | <input checked="" type="checkbox"/> SI Slovenia |
| <input checked="" type="checkbox"/> FI Finland | <input checked="" type="checkbox"/> SK Slovakia |
| <input checked="" type="checkbox"/> GB United Kingdom | <input checked="" type="checkbox"/> SL Sierra Leone |
| <input checked="" type="checkbox"/> GD Grenada | <input checked="" type="checkbox"/> TJ Tajikistan |
| <input checked="" type="checkbox"/> GE Georgia | <input checked="" type="checkbox"/> TM Turkmenistan |
| <input checked="" type="checkbox"/> GH Ghana | <input checked="" type="checkbox"/> TR Turkey |
| <input checked="" type="checkbox"/> GM Gambia | <input checked="" type="checkbox"/> TT Trinidad and Tobago |
| <input checked="" type="checkbox"/> HR Croatia | <input checked="" type="checkbox"/> TZ United Republic of Tanzania |
| <input checked="" type="checkbox"/> HU Hungary | <input checked="" type="checkbox"/> UA Ukraine |
| <input checked="" type="checkbox"/> ID Indonesia | <input checked="" type="checkbox"/> UG Uganda |
| <input checked="" type="checkbox"/> IL Israel | <input checked="" type="checkbox"/> US United States of America |
| <input checked="" type="checkbox"/> IN India | |
| <input checked="" type="checkbox"/> IS Iceland | |
| <input checked="" type="checkbox"/> JP Japan | <input checked="" type="checkbox"/> UZ Uzbekistan |
| <input checked="" type="checkbox"/> KE Kenya | <input checked="" type="checkbox"/> VN Viet Nam |
| <input checked="" type="checkbox"/> KG Kyrgyzstan | <input checked="" type="checkbox"/> YU Yugoslavia |
| <input checked="" type="checkbox"/> KP Democratic People's Republic of Korea | <input checked="" type="checkbox"/> ZA South Africa |
| | <input checked="" type="checkbox"/> ZW Zimbabwe |

Check-boxes reserved for designating States which have become party to the PCT after issuance of this sheet:

- ☐
- ☐

Precautionary Designation Statement: In addition to the designations made above, the applicant also makes under Rule 4.9(b) all other designations which would be permitted under the PCT except any designation(s) indicated in the Supplemental Box as being excluded from the scope of this statement. The applicant declares that those additional designations are subject to confirmation and that any designation which is not confirmed before the expiration of 15 months from the priority date is to be regarded as withdrawn by the applicant at the expiration of that time limit. (Confirmation (including fees) must reach the receiving Office within the 15-month time limit.)

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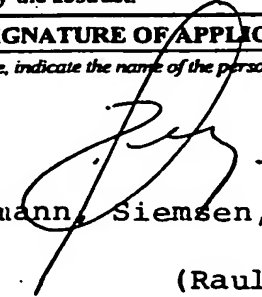
Box N . VI PRIORITY CLAIM					<input type="checkbox"/> Further priority claims are indicated in the Supplemental Box.
Filing date of earlier application (day/month/year)	Number of earlier application	Where earlier application is:			
		national application: country	regional application: regional Office	international application: receiving Office	
item (1) 23 June 1999 (23.06.99)	PI 9902607-4	BR			
item (2)					
item (3)					

☒ The receiving Office is requested to prepare and transmit to the International Bureau a certified copy of the earlier application(s) (only if the earlier application was filed with the Office which for the purposes of the present international application is the receiving Office) identified above as item(s): I

** Where the earlier application is an ARIPO application, it is mandatory to indicate in the Supplemental Box at least one country party to the Paris Convention for the Protection of Industrial Property for which that earlier application was filed (Rule 4.10(b)(ii)). See Supplemental Box.*

Box N . VII INTERNATIONAL SEARCHING AUTHORITY			
Choice of International Searching Authority (ISA) (if two or more International Searching Authorities are competent to carry out the international search, indicate the Authority chosen; the two-letter code may be used):	Request to use results of earlier search; reference to that search (if an earlier search has been carried out by or requested from the International Searching Authority): Date (day/month/year) Number Country (or regional Office)		
ISA/ EPO			

Box No. VIII CHECK LIST: LANGUAGE OF FILING	
This international application contains the following number of sheets: request : 04 description (excluding sequence listing part) : 28 claims : 04 abstract : 01 drawings : 06 sequence listing part of description : - Total number of sheets : 43	This international application is accompanied by the item(s) marked below: 1. <input checked="" type="checkbox"/> fee calculation sheet 2. <input checked="" type="checkbox"/> separate signed power of attorney 3. <input type="checkbox"/> copy of general power of attorney; reference number, if any: 4. <input type="checkbox"/> statement explaining lack of signature 5. <input type="checkbox"/> priority document(s) identified in Box No. VI as item(s): 6. <input type="checkbox"/> translation of international application into (language): 7. <input type="checkbox"/> separate indications concerning deposited microorganism or other biological material 8. <input type="checkbox"/> nucleotide and/or amino acid sequence listing in computer readable form 9. <input checked="" type="checkbox"/> other (specify): assignment document
Figure of the drawings which should accompany the abstract: <u>2a</u>	Language of filing of the international application: <u>English</u>

Box No. IX SIGNATURE OF APPLICANT OR AGENT	
Next to each signature, indicate the name of the person signing and the capacity in which the person signs (if such capacity is not obvious from reading the request).	
 Dannemann, Siemsen, Bigler & Ipanema Moreira (Raul Hey)	

For receiving Office use only	
1. Date of actual receipt of the purported international application: <u>23 JUN 2000 23-6-00</u>	2. Drawings: <input type="checkbox"/> received: <input type="checkbox"/> not received:
3. Corrected date of actual receipt due to later but timely received papers or drawings completing the purported international application:	
4. Date of timely receipt of the required corrections under PCT Article 11(2):	
5. International Searching Authority (if two or more are competent): <u>ISA/</u>	6. <input type="checkbox"/> Transmittal of search copy delayed until search fee is paid.

Date of receipt of the record copy by the International Bureau:	For International Bureau use only
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This sheet is not part of and does not count as a sheet of the international application.

PCT

FEE CALCULATION SHEET Annex to the Request

For receiving Office use only

International Application No.

PCT/RO/00/00005

Applicant's or agent's
file reference

PE-3849

Date stamp of the receiving Office

Applicant

RM MATERIAIS REFRAATÓRIOS LTDA.

CALCULATION OF PRESCRIBED FEES

1. TRANSMITTAL FEE 236,00 T

2. SEARCH FEE DEM. 462,07 X 0.876363 404,94 S

International search to be carried out by EPO
(If two or more International Searching Authorities are competent in relation to the international application, indicate the name of the Authority which is chosen to carry out the international search.)

INTERNATIONAL FEE

Basic Fee

The international application contains 43 sheets.

first 30 sheets CHF 650 x 1.10502 718,26 b1

13 x CHF 15 x 1.10502 = 204,78 b2
remaining sheets additional amount

Add amounts entered at b1 and b2 and enter total at B 923,04 B

Designation Fees

The international application contains 83 designations.

8 x CHF 140 x 1.10502 = 1.237,62 D
number of designation fees amount of designation fee payable (maximum 8)

Add amounts entered at B and D and enter total at I 2.160,66 I

(Applicants from certain States are entitled to a reduction of 75% of the international fee. Where the applicant is (or all applicants are) so entitled, the total to be entered at I is 25% of the sum of the amounts entered at B and D.)

4. FEE FOR PRIORITY DOCUMENT (if applicable) 75,00 P

5. TOTAL FEES PAYABLE R\$ 2.876,60

Add amounts entered at T, S, I and P, and enter total in the TOTAL box

TOTAL

☐ The designation fees are not paid at this time.

MODE OF PAYMENT

☐ authorization to charge
deposit account (see below)

☐ cheque

☐ postal money order

☐ bank draft

☒ cash

☐ revenue stamps

☐ coupons

☐ other (specify):

DEPOSIT ACCOUNT AUTHORIZATION (this mode of payment may not be available at all receiving Offices)

The RO/ ☐ is hereby authorized to charge the total fees indicated above to my deposit account.

☐ (this check-box may be marked only if the conditions for deposit accounts of the receiving Office so permit) is hereby authorized to charge any deficiency or credit any overpayment in the total fees indicated above to my deposit account.

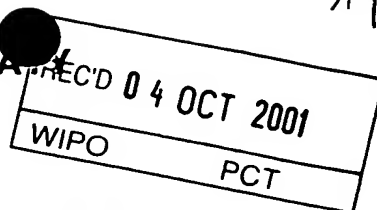
☐ is hereby authorized to charge the fee for preparation and transmittal of the priority document to the International Bureau of WIPO to my deposit account.

Deposit Account No.

Date (day/month/year)

Signature

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INTERNATIONAL PRELIMINARY EXAMINATION REPORT



(PCT Article 36 and Rule 70)

Applicant's or agent's file reference PE-3849	FOR FURTHER ACTION See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)	
International application No. PCT/BR00/00065	International filing date (day/month/year) 23/06/2000	Priority date (day/month/year) 23/06/1999
International Patent Classification (IPC) or national classification and IPC B01J19/02		
Applicant RM MATERIAIS REFRATARIOS LTDA. et al.		

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.
2. This REPORT consists of a total of 6 sheets, including this cover sheet.
☐ This report is also accompanied by ANNEXES, i.e. sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).
These annexes consist of a total of sheets.

3. This report contains indications relating to the following items:

- I ☒ Basis of the report
- II ☐ Priority
- III ☐ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
- IV ☒ Lack of unity of invention
- V ☒ Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
- VI ☐ Certain documents cited
- VII ☐ Certain defects in the international application
- VIII ☒ Certain observations on the international application

Date of submission of the demand 22/01/2001	Date of completion of this report 02.10.2001
Name and mailing address of the international preliminary examining authority:  European Patent Office D-80298 Munich Tel. +49 89 2399 - 0 Tx: 523656 epmu d Fax: +49 89 2399 - 4465	Authorized officer Buesing, G Telephone No. +49 89 2399 8356 

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INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No. PCT/BR00/00065

I. Basis of the report

1. With regard to the **elements** of the international application (*Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report since they do not contain amendments (Rules 70.16 and 70.17)*):

Description, pages:

1-28 as originally filed

Claims, No.:

1-25 as originally filed

Drawings, sheets:

1/6-6/6 as originally filed

2. With regard to the **language**, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.

These elements were available or furnished to this Authority in the following language: , which is:

- ☐ the language of a translation furnished for the purposes of the international search (under Rule 23.1(b)).
- ☐ the language of publication of the international application (under Rule 48.3(b)).
- ☐ the language of a translation furnished for the purposes of international preliminary examination (under Rule 55.2 and/or 55.3).

3. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

- ☐ contained in the international application in written form.
- ☐ filed together with the international application in computer readable form.
- ☐ furnished subsequently to this Authority in written form.
- ☐ furnished subsequently to this Authority in computer readable form.
- ☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.
- ☐ The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

4. The amendments have resulted in the cancellation of:

- ☐ the description, pages:
- ☐ the claims, Nos.:

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**INTERNATIONAL PRELIMINARY
EXAMINATION REPORT**

International application No. PCT/BR00/00065

☐ the drawings, sheets:

5. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)):

(Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report.)

6. Additional observations, if necessary:

IV. Lack of unity of invention

1. In response to the invitation to restrict or pay additional fees the applicant has:

- ☐ restricted the claims.
☒ paid additional fees.
☐ paid additional fees under protest.
☐ neither restricted nor paid additional fees.

2. ☐ This Authority found that the requirement of unity of invention is not complied and chose, according to Rule 68.1, not to invite the applicant to restrict or pay additional fees.

3. This Authority considers that the requirement of unity of invention in accordance with Rules 13.1, 13.2 and 13.3 is

- ☐ complied with.
☒ not complied with for the following reasons:
see separate sheet

4. Consequently, the following parts of the international application were the subject of international preliminary examination in establishing this report:

- ☒ all parts.
☐ the parts relating to claims Nos. .

V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Yes:	Claims
	No:	Claims 1
Inventive step (IS)	Yes:	Claims 15-25
	No:	Claims 2-14

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**INTERNATIONAL PRELIMINARY
EXAMINATION REPORT**

International application No. PCT/BR00/00065

Industrial applicability (IA) Yes: Claims 1-25
 No: Claims

2. Citations and explanations
 see separate sheet

VIII. Certain observations on the international application

The following observations on the clarity of the claims, description, and drawings or on the question whether the claims are fully supported by the description, are made:
see separate sheet

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Re Item IV

Lack of unity of invention

1. Independent claim 1 discloses an apparatus comprising an external and an internal coating and a space between these two coatings adapted to be under vacuum. The dependent claims 2 - 14 disclose further details of the apparatus.
2. Independent claim 15 discloses a biomass pre-hydrolysis process which is carried out under rotary oscillation of the apparatus wherein the reaction is carried out. The dependent claims 16 - 25 disclose further details of the process.
3. As there is no common concept linking the groups of inventions, lack of unity of invention is notified.

Re Item V

Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

A. Invention 1 (claims 1-14):

1. Claim 1 generally defines an apparatus comprising internal and external coatings which are separated by a vacuum space. No particular constructional features of the apparatus are defined in the independent claim 1. However, the apparatus should be suitable for the pre-hydrolysis of biomass.
2. Reference is made to the following documents:

D1: US-A-3 056 664
D2: US-A-4 997 124
3. Both D1 and D2 disclose an apparatus comprising an external coating (D1: 2; D2: 2), an internal coating (D1:16; D2:3) covering the inside of the external coating. The prior art apparatus further comprise a space between the internal coating and the external coating wherein a vacuum is produced (D1: 10; D2:5). Therefore, both D1 and D2 disclose all the constructional features defined in claim 1. As it

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also appears to be possible to pre-hydrolyse biomass in the apparatus according to D1 and D2, the intended use mentioned in claim 1 cannot support novelty. Consequently, the subject-matter of claim 1 lacks novelty.

4. It is not apparent how the features of the dependent claims 2-14 could support an inventive step. Therefore, it is not seen which part of the application could serve as a basis for a claim defining new and inventive subject-matter.

B. Invention 2 (claims 15-25):

5. Claim 15 discloses a biomass pre-hydrolysis process which is carried out under rotary oscillation of the apparatus wherein the reaction is carried out. The available prior art documents do neither disclose nor suggest to carry out such a process under an oscillatory rotation. Therefore, both novelty and an inventive step are acknowledged for the process claims.

Re Item VIII

Certain observations on the international application

1. Some of the claims lack clarity:

Claim 6: The definition of the apparatus as a reactor does not add structural features to the claimed subject-matter. It appears that claim 6 is superfluous.

Claims 12, 13: These claims lack clarity because the features mentioned therein are not suitable to further define the claimed apparatus. They rather relate to process steps which are suitable when the reactor is used.

Claim 15: It is not clear what is meant by the unit "bricks" in feature d). This applies also to page 22 of the description.

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PATENT COOPERATION TREATY

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Patent Cooperation Treaty
BIGGER & MOREIRA

From the
INTERNATIONAL PRELIMINARY EXAMINING AUTHORITY

PCT

NOTIFICATION OF TRANSMITTAL OF THE INTERNATIONAL PRELIMINARY EXAMINATION REPORT (PCT Rule 71.1)

To:

DANNEMANN, SIEMSEN, BIGLER &
IPANEMA MOREIRA
Rua Marquês de Olinda 70
Caixa Postal 2142
Botafogo
22251-040- Rio de Janeiro - RJ
BRESIL

Date of mailing
(day/month/year) 02.10.2001

Applicant's or agent's file reference
PE-3849

IMPORTANT NOTIFICATION

International application No.
PCT/BR00/00065

International filing date (day/month/year)
23/06/2000

Priority date (day/month/year)
23/06/1999

Applicant
RM MATERIAIS REFRATARIOS LTDA. et al.

1. The applicant is hereby notified that this International Preliminary Examining Authority transmits herewith the international preliminary examination report and its annexes, if any, established on the international application.
2. A copy of the report and its annexes, if any, is being transmitted to the International Bureau for communication to all the elected Offices.
3. Where required by any of the elected Offices, the International Bureau will prepare an English translation of the report (but not of any annexes) and will transmit such translation to those Offices.

4. REMINDER

The applicant must enter the national phase before each elected Office by performing certain acts (filing translations and paying national fees) within 30 months from the priority date (or later in some Offices) (Article 39(1)) (see also the reminder sent by the International Bureau with Form PCT/IB/301).

Where a translation of the international application must be furnished to an elected Office, that translation must contain a translation of any annexes to the international preliminary examination report. It is the applicant's responsibility to prepare and furnish such translation directly to each elected Office concerned.

For further details on the applicable time limits and requirements of the elected Offices, see Volume II of the PCT Applicant's Guide.

Name and mailing address of the IPEA/

European Patent Office
D-80298 Munich
Tel. +49 89 2399 - 0 Tx: 523656 epmu d
Fax: +49 89 2399 - 4465

Authorized officer

Ferro Vasconcelos, M

Tel. +49 89 2399-7995 8041





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INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference PE-3849		FOR FURTHER ACTION See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)	
International application No. PCT/BR00/00065	International filing date (day/month/year) 23/06/2000	Priority date (day/month/year) 23/06/1999	
International Patent Classification (IPC) or national classification and IPC B01J19/02			
Applicant RM MATERIAIS REFRATARIOS LTDA. et al.			
<p>1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.</p> <p>2. This REPORT consists of a total of 6 sheets, including this cover sheet.</p> <p><input type="checkbox"/> This report is also accompanied by ANNEXES, i.e. sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).</p> <p>These annexes consist of a total of sheets.</p>			
<p>3. This report contains indications relating to the following items:</p> <ul style="list-style-type: none"> I <input checked="" type="checkbox"/> Basis of the report II <input type="checkbox"/> Priority III <input type="checkbox"/> Non-establishment of opinion with regard to novelty, inventive step and industrial applicability IV <input checked="" type="checkbox"/> Lack of unity of invention V <input checked="" type="checkbox"/> Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement VI <input type="checkbox"/> Certain documents cited VII <input type="checkbox"/> Certain defects in the international application VIII <input checked="" type="checkbox"/> Certain observations on the international application 			
Date of submission of the demand 22/01/2001		Date of completion of this report 02.10.2001	
Name and mailing address of the international preliminary examining authority:  European Patent Office D-80298 Munich Tel. +49 89 2399 - 0 Tx: 523656 epmu d Fax: +49 89 2399 - 4465		Authorized officer Buesing, G Telephone No. +49 89 2399 8356 	

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INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No. PCT/BR00/00065

I. Basis of the report

1. With regard to the elements of the international application (*Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report since they do not contain amendments (Rules 70.16 and 70.17)*):
- Description, pages:**

1-28 as originally filed

Claims, No.:

1-25 as originally filed

Drawings, sheets:

1/6-6/6 as originally filed

2. With regard to the **language**, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.

These elements were available or furnished to this Authority in the following language: , which is:

- ☐ the language of a translation furnished for the purposes of the international search (under Rule 23.1(b)).
- ☐ the language of publication of the international application (under Rule 48.3(b)).
- ☐ the language of a translation furnished for the purposes of international preliminary examination (under Rule 55.2 and/or 55.3).

3. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

- ☐ contained in the international application in written form.
- ☐ filed together with the international application in computer readable form.
- ☐ furnished subsequently to this Authority in written form.
- ☐ furnished subsequently to this Authority in computer readable form.
- ☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.
- ☐ The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

4. The amendments have resulted in the cancellation of:

- ☐ the description, pages:
- ☐ the claims, Nos.:

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**INTERNATIONAL PRELIMINARY
EXAMINATION REPORT**

International application No. PCT/BR00/00065

☐ the drawings, sheets:

5. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)):

(Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report.)

6. Additional observations, if necessary:

IV. Lack of unity of invention

1. In response to the invitation to restrict or pay additional fees the applicant has:

- ☐ restricted the claims.
☒ paid additional fees.
☐ paid additional fees under protest.
☐ neither restricted nor paid additional fees.

2. ☐ This Authority found that the requirement of unity of invention is not complied and chose, according to Rule 68.1, not to invite the applicant to restrict or pay additional fees.

3. This Authority considers that the requirement of unity of invention in accordance with Rules 13.1, 13.2 and 13.3 is

- ☐ complied with.
☒ not complied with for the following reasons:
see separate sheet

4. Consequently, the following parts of the international application were the subject of international preliminary examination in establishing this report:

- ☒ all parts.
☐ the parts relating to claims Nos. .

V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Yes:	Claims
	No:	Claims 1
Inventive step (IS)	Yes:	Claims 15-25
	No:	Claims 2-14

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Re Item IV

Lack of unity of invention

1. Independent claim 1 discloses an apparatus comprising an external and an internal coating and a space between these two coatings adapted to be under vacuum. The dependent claims 2 - 14 disclose further details of the apparatus.
2. Independent claim 15 discloses a biomass pre-hydrolysis process which is carried out under rotary oscillation of the apparatus wherein the reaction is carried out. The dependent claims 16 - 25 disclose further details of the process.
3. As there is no common concept linking the groups of inventions, lack of unity of invention is notified.

Re Item V

Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

A. Invention 1 (claims 1-14):

1. Claim 1 generally defines an apparatus comprising internal and external coatings which are separated by a vacuum space. No particular constructional features of the apparatus are defined in the independent claim 1. However, the apparatus should be suitable for the pre-hydrolysis of biomass.
2. Reference is made to the following documents:

D1: US-A-3 056 664
D2: US-A-4 997 124
3. Both D1 and D2 disclose an apparatus comprising an external coating (D1: 2; D2: 2), an internal coating (D1:16; D2:3) covering the inside of the external coating. The prior art apparatus further comprise a space between the internal coating and the external coating wherein a vacuum is produced (D1: 10; D2:5). Therefore, both D1 and D2 disclose all the constructional features defined in claim 1. As it

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**INTERNATIONAL PRELIMINARY
EXAMINATION REPORT - SEPARATE SHEET**

International application No. PCT/BR00/00065

also appears to be possible to pre-hydrolyse biomass in the apparatus according to D1 and D2, the intended use mentioned in claim 1 cannot support novelty. Consequently, the subject-matter of claim 1 lacks novelty.

4. It is not apparent how the features of the dependent claims 2-14 could support an inventive step. Therefore, it is not seen which part of the application could serve as a basis for a claim defining new and inventive subject-matter.

B. Invention 2 (claims 15-25):

5. Claim 15 discloses a biomass pre-hydrolysis process which is carried out under rotary oscillation of the apparatus wherein the reaction is carried out. The available prior art documents do neither disclose nor suggest to carry out such a process under an oscillatory rotation. Therefore, both novelty and an inventive step are acknowledged for the process claims.

Re Item VIII

Certain observations on the international application

1. Some of the claims lack clarity:

Claim 6: The definition of the apparatus as a reactor does not add structural features to the claimed subject-matter. It appears that claim 6 is superfluous.

Claims 12, 13: These claims lack clarity because the features mentioned therein are not suitable to further define the claimed apparatus. They rather relate to process steps which are suitable when the reactor is used.

Claim 15: It is not clear what is meant by the unit "bricks" in feature d). This applies also to page 22 of the description.

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INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No. PCT/BR00/00065

Industrial applicability (IA) Yes: Claims 1-25
 No: Claims

2. Citations and explanations
see separate sheet

VIII. Certain observations on the international application

The following observations on the clarity of the claims, description, and drawings or on the question whether the claims are fully supported by the description, are made:
see separate sheet

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